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Biosorption of Zinc on Palm Tree Leaves: Equilibrium, Kinetics, and Thermodynamics Studies

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Abstract: The efficiency of using palm tree leaves to remove zinc ions from aqueous solution was studied. Adsorption isotherms, kinetics, and thermodynamics studies were conducted. The influence of different experimental parameters, such as equilibrium pH, shaking rate, temperature, and the presence of other pollutants such as chelating agents on the biosorption of zinc on palm tree leaves was investigated.

Batch biosorption experiments showed that palm tree leaves used in this study proved to be suitable for the removal of zinc from dilute solutions where a maximum uptake capacity of 14.7 mg/g was obtained at 25°C. Zinc biosorption on palm tree leaves was found to be highly pH dependent. The biosorption process was found to be rapid with 90% of the adsorption completed in about 10 min. Dynamics studies of the biosorption of zinc on palm tree leaves showed that the biosorption process followed the pseudo second-order kinetics with little intraparticle diffusion mechanism contribution. The equilibrium results indicated that zinc biosorption on palm tree leaves could be described by the Langmuir, Freundlich, Gin et al., and Sips models. Using the Langmuir equilibrium constants obtained at different temperatures, the thermodynamics properties of the biosorption (ΔG^0 , ΔH^0 , and ΔS^0) were also determined. The values of these parameters indicated the spontaneous and endothermic nature of zinc biosorption on palm tree leaves.

Keywords: Biosorption, zinc, palm tree leaves, isotherms

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INTRODUCTION

The last century witnessed accelerated and intensified industrialization and urbanization activities which led to degradation and contamination of our aquatic environment with different serious pollutants. Heavy metals such as zinc, copper, lead, and nickel are among the most common inorganic pollutants found in industrial effluents. These metals pose serious environmental problems due to their toxicity, even at low concentrations, and their ability to accumulate throughout the food chain via bioaccumulation. Zinc is listed by the US Environmental Protection Agency as one of the 129 pollutants found in wastewaters that constitutes serious health hazards. According to the World Health Organization (WHO), the maximum acceptable concentration of zinc in drinking water is 3.0 ppm (1).

Zinc may be found in wastewater discharged from municipal wastewater treatment plants, chemicals, metals, pulp, and paper industries, acid marine drainage and galvanizing plants, viscose rayon yarn and fiber production, etc. Traditional methods for removing zinc ions or other dissolved heavy metals include chemical precipitation, ion exchange carbon, adsorption, evaporation, and membrane processes. However, for wastewaters with heavy metal concentrations less than 100 ppm, these methods are either expensive or ineffective (2–4, 6). Thus, there is a need for a low cost and yet effective treatment method for such liquid wastes.

Research during the last two decades revealed that biosorption, which involves using biological materials as sorbents, is a promising alternative technology for treatment of municipal or industrial wastewaters for the separation and recovery of heavy metal ions. This treatment method is based on utilizing the ability of biological materials to accumulate heavy metals from liquid wastes by either metabolically mediated, or physico-chemical pathways of uptake. These biological materials include microorganisms (4–11), agricultural wastes (12–16), and animal wastes (2, 3).

Palm trees are abundant all over the world, particularly in the Arab Gulf area. In the United Arab Emirates, it is estimated that there are over ten millions of fruitful palm trees (17). Due to the presence of different functional groups, such as hydroxyl, carboxylic, and phenolic on the leaves of these trees, these leaves are expected to be good heavy metals biosorbents.

The purpose of this study is to investigate the potential use of palm tree leaves for the biosorption of zinc ions. The effects of different parameters such as equilibrium pH, temperature, and shaking time on zinc removal from aqueous solutions will be investigated. The thermodynamics of biosorption of zinc on palm tree leaves will be analyzed. The biosorption equilibrium data will be analyzed using different adsorption isotherm models. The dynamics of biosorption of zinc will be tested using the pseudo-second order kinetics in addition to the Weber and Morris equation. The effect of presence of other pollutants such, EDTA on the biosorption of zinc will be investigated.

MATERIALS AND METHODS

Biosorbent Material

The palm tree leaves used in this study were thoroughly washed by distilled water to remove any impurities, dried, ground, and sieved and then stored in bottles. The functional acidic groups on the palm tree leaves were determined using Boehm's titration method (17) as follows: 0.2 g of the prepared palm tree leaves were dispersed in 50 ml deionized water. The suspension was mixed with 0.1 N solutions of sodium hydroxide, sodium bicarbonate, and sodium carbonate, and then shaken for 48 h at room temperature. After this time, the sample was left for 6 h so that particles could settle. The sample was then filtered and 20 ml of the filtrate was added to 15 ml of 0.1 N HCl. The excess HCl was determined by titration with 0.1 N NaOH. According to this titration method, sodium bicarbonate will neutralize carboxyl groups, sodium carbonate will neutralize carboxyl, lactones, and lactols groups, and sodium hydroxide will neutralize carboxyl, lactones, lactols, and phenols groups. Thus, the functional groups can be identified by the known volumes of acids and bases used in titration. Table 1 lists the acidic functional groups found on the palm tree leaves.

Chemicals

Analytical grade reagents of nitrate of zinc were used to prepare 1000 ppm stock solution of metal ions by diluting the metal nitrate with double distilled water. Nitrate was chosen as the counter ion because of its low tendency to form metal complexes. The pH was adjusted by dilute nitric acid and dilute sodium hydroxide solutions.

Batch Single Heavy Metal Biosorption Experiments

Batch biosorption assays were carried out in 100 ml bottles by mixing 50 ml of the metal solution of the desired concentration and amount of sorbent in

Table 1. The functional groups on the palm tree leaves used in this study

Functional group	Meq H ⁺ /g palm tree leaves
Carboxyl	0.85
Lactones and lactols	2.8
Phenols	0.65

100 ml bottles. Equilibrium isotherms were obtained by agitating 0.10 g of palm tree leaves with 50 ml of zinc ion solution for a predetermined time in a water bath shaker at 25°C. For adsorption isotherm studies, different zinc ion concentrations were used (20–300 ppm). Dynamics studies were carried out with 100 ppm initial zinc ions concentration, and mass of palm tree leaves of 0.10 g. Samples were taken at periodic intervals and then, the zinc solution was separated from the sorbent by filtration and the concentration of zinc was determined using a Verian atomic absorption spectrophotometer.

Material balance on zinc can be used to determine the amount of zinc removed by the biomass. Zinc uptake, or the amount of zinc removed per unit mass of the palm tree leaves can be calculated using the following equation:

$$q = \frac{(C_0 - C_e)V}{w} \quad (1)$$

where q is the amount of zinc adsorbed by biomass (mg/g), C_0 the initial concentration of zinc (mg/L), C_e the concentration of zinc at equilibrium (mg/L), V the initial volume of zinc solution (L), and w is the mass of the palm tree leaves (g).

Batch Competitive Biosorption Experiments

The competitive biosorption of zinc with EDTA was also investigated. The studies were carried out at constant initial zinc ion concentration of 100 ppm and varying EDTA initial concentration. All the experiments were conducted at 25°C, and using the same procedures used in the single ion adsorption experiments.

RESULTS AND DISCUSSION

Effect of Biosorbent Dose

The effect of biomass dose on the removal of zinc was studied at 5.5 pH, 100 ppm initial zinc concentration and 25°C, and the results are presented in Fig. 1. Figure 1 shows that increasing biomass dose increases the zinc percent removal until an optimum value of biomass dose, 0.10 g, is reached, and beyond this value there will be no significant increase in the percent removal of zinc with the increase in biomass dose. The increase in zinc percent removal with increasing biomass dose is due to the fact that by increasing the biomass dose, the number of active binding sites on the biosorbent increases, thus the percent removal increases. However, Fig. 1 shows that zinc uptake decreases with the increase in biosorbent dose. The decrease in metal uptake by biosorbent dose can be attributed to many factors, such as

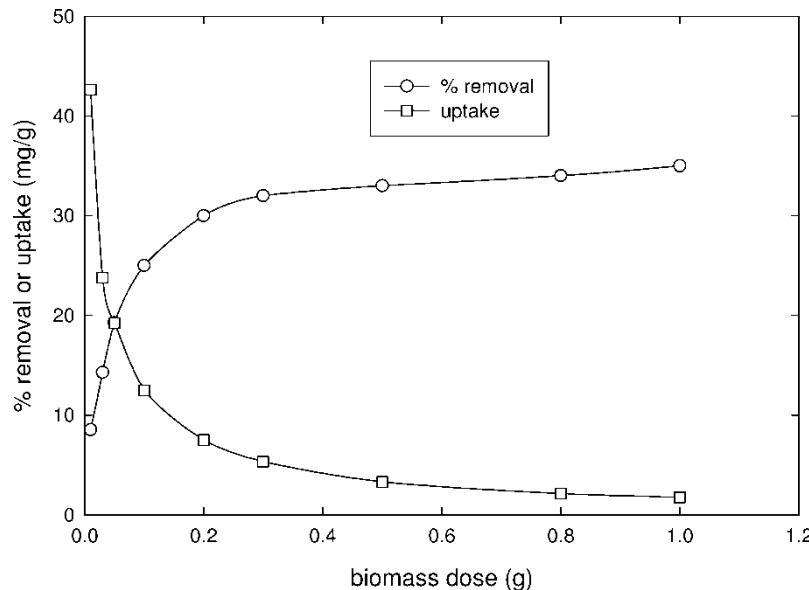


Figure 1. Effect of biomass dose on zinc removal (Initial zinc ions concentration = 100 ppm, $T = 25^\circ\text{C}$, $\text{pH} = 5.5$, time = 24.0 hr).

the possibility of poor mixing at higher biomass concentration, electrostatic interactions, and interference between binding sites (4, 6, 18, 19). These factors result in lower adsorptive capacity utilization, and hence decreased adsorption efficiency. The above results are important in the design of batch adsorption processes since they suggest that using small batches of sorbent is more economical for the removal of heavy metal ions (20, 21).

Effect of pH

Biosorption processes are strongly pH dependent. Variation of pH changes the surface charges on biosorbents which will affect the ion exchange; one of the biosorption mechanisms, and thus the pH reflects the nature of the physico-chemical interaction of species in solution and the surface adsorptive sites. Different species may have different pH optima, possibly due to the different solution chemistry of the species (4, 6, 20, 22).

Figure 2 shows the effect of pH on the biosorption of zinc ions on palm tree leaves. As can be depicted from this figure, the biosorption of zinc was strongly affected by equilibrium pH, where insignificant uptake was obtained at pH below 4.0, while sharp increase in zinc uptake was observed at pH 4.0. No significant increase in zinc uptake was observed at pH values between 5.0–5.8. At low pH values, the surface of the biosorbent would be

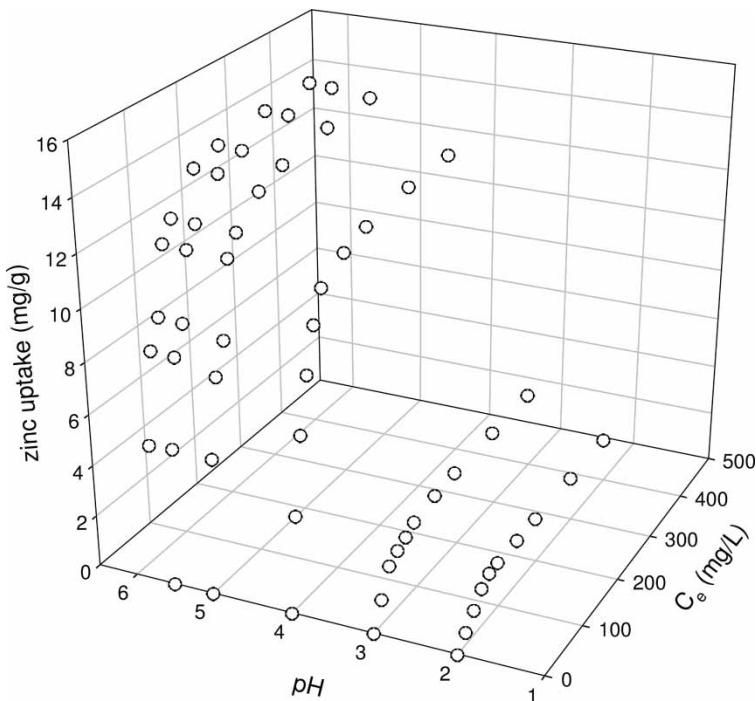


Figure 2. Effect of pH on zinc removal (Initial zinc ions concentration = 100 ppm, T = 25°C, concentration of palm tree leaves = 2.0 mg/ml).

closely associated with hydronium ions which impede the access of metal ions to the surface functional groups and consequently decrease the metal percentage removal (4, 6). Increasing pH involves less hydronium ions and hence more ligands will be available for metal ion binding, which enhances the biosorption process. At low pH (e.g., pH < 6.0), zinc and most of the heavy metals are present in their ionic state (22, 23), then the sharp increase in zinc biosorption from pH 4 to 5.5 cannot be attributed to the change in zinc speciation.

Adsorption Isotherms

Adsorption equilibrium isotherms, shown in Fig. 3, describe the distribution of metal ions between the solid and the metal solution at equilibrium. These isotherms are key factors in the design of adsorption systems. There are many isotherm models that can be used to analyze adsorption isotherms. Langmuir isotherm model is considered one of the most widely used models to analyze adsorption equilibrium isotherms. This model is valid for

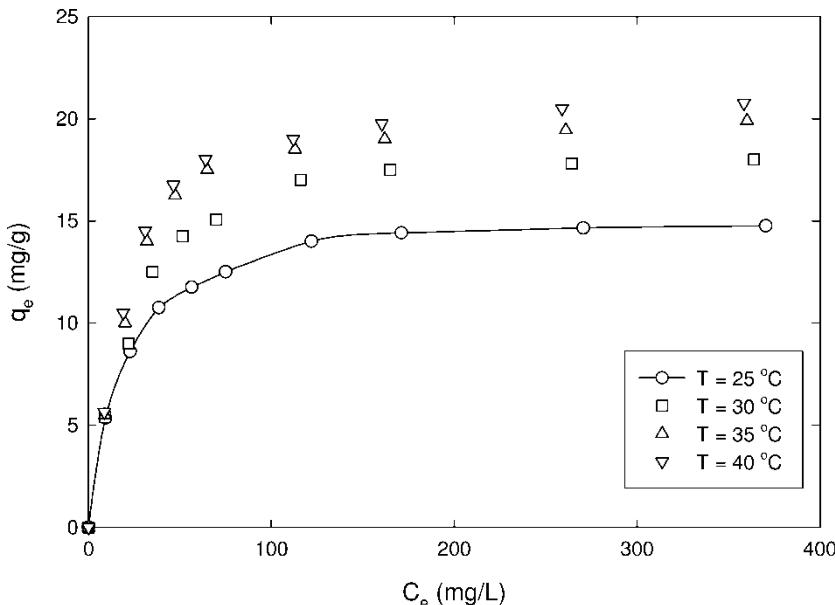


Figure 3. Experimental isotherms of zinc ions biosorbed on palm tree leaves (pH = 5.5 ppm, concentration of palm tree leaves = 2.0 mg/ml).

monolayer sorption onto a surface with a finite number of identical sites and uniform adsorption energies where there are interactions between the adsorbed molecules. In its simplest form, Langmuir isotherm is given by the equation

$$q_e = \frac{q_{mon} K_L C_e}{1 + K_L C_e} \quad (2)$$

where q_{mon} and K_L are the Langmuir constants which are related to the amount of adsorption corresponding to monolayer coverage, or adsorption capacity, and the energy of adsorption, respectively. These constants can be determined by linearizing Eq. (2). One of the most widely used linearized forms of Eq. (2) is the form which yields a straight line by plotting C_e/q_e against C_e , as shown in Fig. 4. The Langmuir parameters for the biosorption of zinc on palm tree leaves were found from the slopes and intercepts of the straight line shown in Fig. 4 and are listed in Table 2. Figure 4 and the value of the correlation coefficient of the linear regression ($R^2 = 0.99$) indicate that the Langmuir model could describe the biosorption of zinc on palm tree leaves. The value of adsorption capacity, or maximum uptake; q_{mon} , can be used to compare the efficiency of different adsorbents, as shown in Table 3 which shows a comparison of different adsorbents for zinc removal. As can be noticed

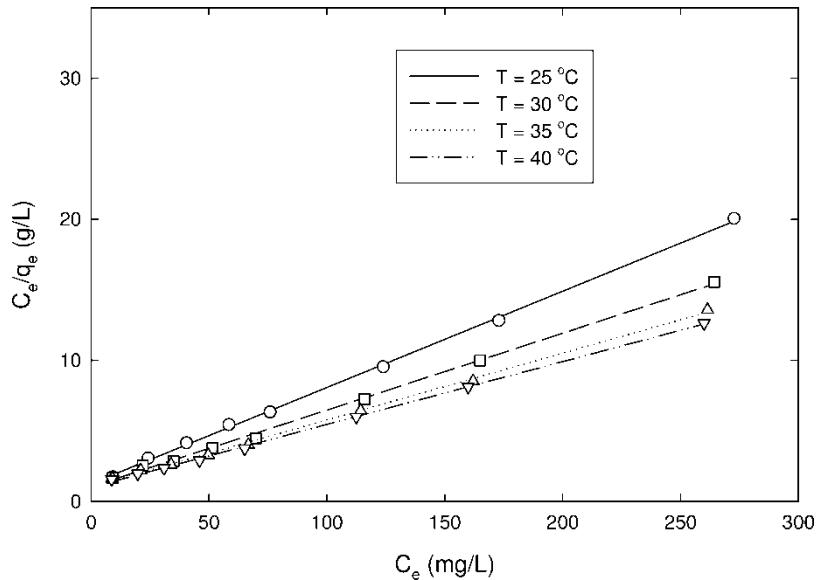


Figure 4. Langmuir isotherms of zinc ions biosorbed on palm tree leaves (pH = 5.5 ppm, concentration of palm tree leaves = 2.0 mg/ml).

from this table, palm tree leaves are among the efficient adsorbent for zinc removal.

The Freundlich isotherm model, which is an empirical equation, is another widely used isotherm to describe adsorption data. This model is based on the assumption that sorption occurs on heterogeneous surfaces.

Table 2. Adsorption isotherms parameters for the sorption of zinc on palm tree leaves at T = 25°C

Model	Parameter	Value
Langmuir	q_{mon} (mg/g)	14.6
	K_L (l/mg)	0.056
Freundlich	K (l/mg) ^{1/n}	3.01
	(mg/g)	
Gin et al.	n	3.23
	α	0.896
Sips	β (mg/L · g)	-139.718
	q_{mon} (mg/g)	16.0
	K_L (l/mg)	0.05
	n	1.06
	O.F (mg/g) ²	1.9

Table 3. Adsorption capacity for zinc using different adsorbents

Adsorbent	Adsorption capacity (mg/g) of Zn ⁺²	Reference
Hazelnut shells	1.78	(24)
Peat	9.3	(25)
Activated carbon prepared from almond shells	6.65	(26)
Commercial activated carbon	19.9	(27)
Palm tree leaves	14.7	This study

However, it does not provide any information on the monolayer adsorption capacity. The simplest form of this model is given by the equation

$$q_e = KC_e^{1/n} \quad (3)$$

where K and n are the Freundlich constants which are indicators of adsorption capacity and adsorption intensity, respectively. Equation (3) can be linearized to determine the Freundlich parameters; K_F and n , by plotting $\ln q_e$ vs. $\ln C_e$. The Freundlich equation could fit the data of the biosorption of zinc on palm tree leaves with the correlation coefficient of the linear regression $R^2 = 0.975$. The values of the Freundlich parameters are listed in Table 2. As can be noticed, the value of n was greater than 1, which also indicates favorable biosorption of zinc on palm tree leaves.

Gin et al. (28) proposed a simplified equilibrium model given by the equation

$$\frac{C_e}{C_0} = \alpha \exp\left(\beta \frac{w}{C_0}\right) \quad (4)$$

where α and β are the parameters of the proposed model, to describe the biosorption of heavy metals on algae. A key difference between this model and other isotherm models is that metal removal is related to the initial and equilibrium biomass concentrations rather than the equilibrium concentration only. This model is used to describe the biosorption of zinc on palm tree leaves. According to this model, a plot of $\ln (C_e/C_0)$ against $1/C_0$ results in a straight line with a slope of βw and intercept of $\ln(\alpha)$. The parameters of the model found by fitting Eq. (4) are listed in Table 2. The value of the correlation coefficient of the linear regression (R^2) was found to be 0.993, which shows that this model can be used to describe the biosorption of zinc on palm tree leaves. Negative values of β confirm the feasibility of the biosorption process. The absolute value of β can be used to indicate the metal sorption ability of the biosorbent. The higher absolute values of β , the higher values of the slope of the curves, and the higher the sorption ability (28). Gin et al.

argued that under ideal conditions, the value of α should be unity; hence, deviations from unity due to nonideal conditions are expected.

The Sips model, given by the equation (29)

$$q_e = \frac{q_{mon}(K_L C_e)^{1/n}}{1 + (K_L C_e)^{1/n}} \quad (5)$$

where q_{mon} , K_L , and n are the Sips constants, is an example of a three-parameter adsorption isotherm equation that was proposed to improve the fit of the two-parameter adsorption isotherm models. At low sorbate concentration, this model assumes the form of the Freundlich model, while at high concentrations it predicts a constant, monolayer sorption behavior similar to that of the Langmuir isotherm. The experimental data of biosorption of zinc on palm tree leaves were analyzed using the Sips equation. The Sips constants were found by solving the following objective function

$$O.F. = \min \sum_{i=1}^m (q_{e,exp} - q_{e,cal})^2 \quad (6)$$

where m is the number of experimental data. The values of the Sips constants along with the value of O.F. defined in Eq. (6) for the biosorption of zinc on palm tree leaves are listed in Table 2. These values indicate that this equation fits the experimental data adequately.

Effect of Shaking Time and Dynamics of Zinc Biosorption

The effect of shaking time on percent removal of zinc was studied using solutions of zinc with initial concentration of 100 ppm at pH 5.5 and palm tree leaves dose of 2.0 mg/ml. The shaking time was varied from 5 to 300 minutes, and the results are shown in Fig. 5. Figure 5 shows that maximum zinc biosorption on palm tree leaves was reached at 45 minutes, beyond which there was no further increase in zinc biosorption. Thus, 45 minutes shaking time was considered to be adequate for the zinc biosorption on palm tree leaves. Moreover, Fig. 5 indicates that zinc biosorption on palm tree leaves involved two stages. In the first stage, sorption was rapid where approximately 90% of the maximum zinc biosorption occurs within the first 10 minutes. This rapid sorption stage indicates that passive surface sorption through physical adsorption or the biosorbent surface ion exchange occurs on the palm tree leaves surface. The second stage is slow and may involve other adsorption mechanisms such as intraparticle diffusion. Similar trends were obtained for other heavy metals sorbed on other different biosorbents (4, 6). The advantage of such rapid sorption in practical applications is that smaller reactor volumes can be used (4, 6).

Next, the reaction kinetics of biosorption of zinc on palm tree leaves will be analyzed using the pseudo-second order equation. The binding of zinc ions

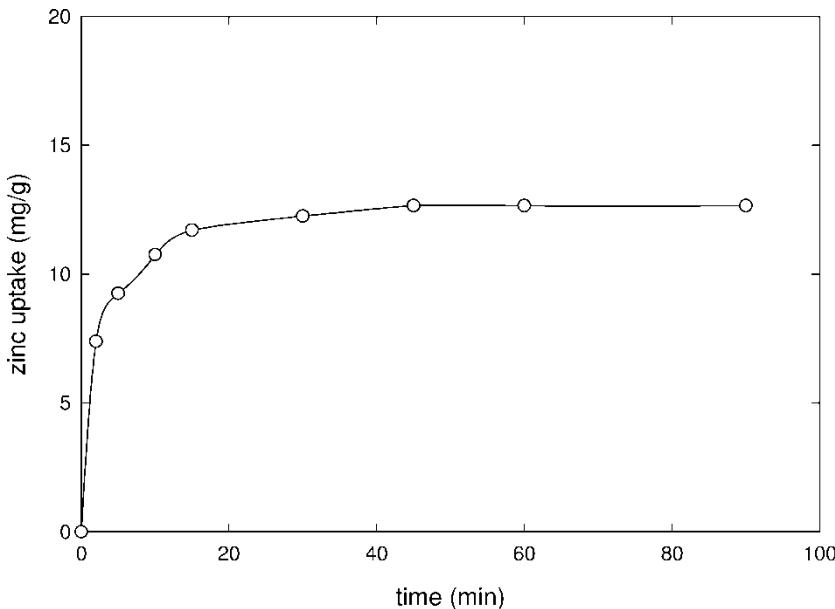
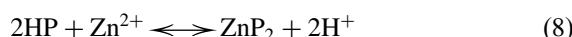


Figure 5. Effects of shaking time on zinc removal (Initial zinc ions concentration = 100 ppm, $T = 25^\circ\text{C}$, $\text{pH} = 5.5$, concentration of palm tree leaves = 2.0 mg/ml).

to the polar sites on palm tree leaves surface; P^- and HP , can be theorized to be according to the following two reactions (30–33)



or



Assuming the sorption capacity of zinc on palm tree leaves is proportional to the number of active sites occupied on the sorbent, and then the pseudo-second order is given by (30–33)

$$\frac{dq_t}{dt} = k(q_e - q_t)^2 \quad (9)$$

where k (g/mg · min) is the equilibrium rate constant of pseudo-second order sorption kinetics, q_t (mg/g) is the amount of sorbate on sorbent at time t and q_e is the equilibrium uptake. Equation (9) can be solved with the boundary condition: $q = 0$ @ $t = 0$, to give

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{q_e^2 k} \quad (10)$$

The values of q_e and k can be calculated from the slopes and the intercepts of the straight line resulting from plotting t/q_t vs. t . The values of q_e and k for the biosorption of zinc ions on palm tree leaves were found to be 12.70 mg/g and 0.046 (g/mg · min), respectively, with the value of $R^2 = 0.999$. These results show that the value of the theoretical q_e is in a good agreement with that found experimentally (12.65 mg/g), and thus the biosorption of zinc on palm tree leaves assumed to follow pseudo-second order kinetics.

The Webber and Morris equation (34) given below can be used to investigate the contribution of intraparticle diffusion mechanism in zinc biosorption on palm tree leaves

$$q_t = k_d t^{0.5} \quad (11)$$

where k_d (mmol/g · h^{-0.5}) is the rate constant of intraparticle diffusion. According to this equation, for intraparticle diffusion mechanism contribution, the plot of q_t vs. $t^{0.5}$ should be linear, and if the straight line passes through the origin, then the intraparticle diffusion mechanism would be the only mechanism involved in the sorption process. The application of this equation to the biosorption of zinc on palm tree leaves over the narrow equilibrium time range (0–20 minutes), as shown in Fig. 6, shows that the linear relationship holds well. However, the straight line does not pass through the origin which indicates that the intraparticle diffusion is not the only

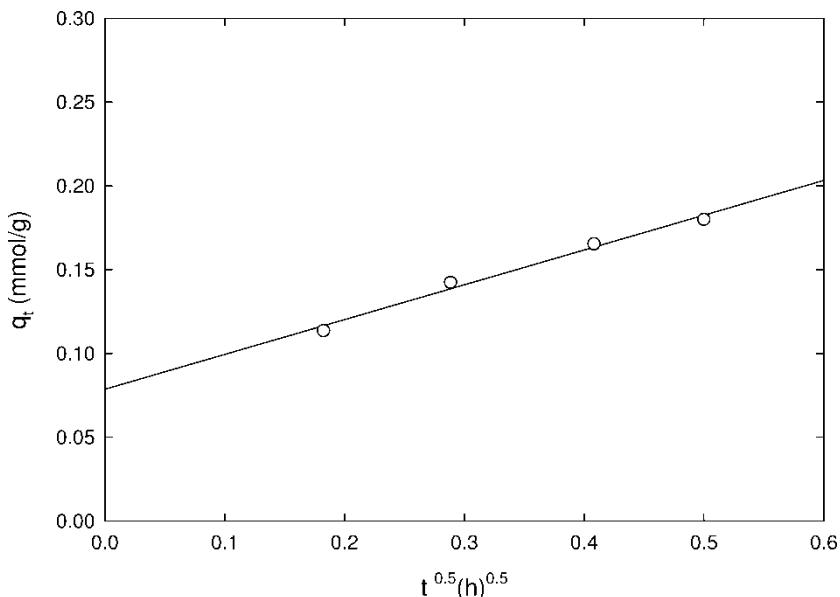


Figure 6. Dynamics of sorption of zinc on palm tree leaves: Application of Webber and Morris equation (Initial zinc ions concentration = 100 ppm, T = 25°C, pH = 5.5, concentration of palm tree leaves = 2.0 mg/ml).

mechanism involved in the biosorption of zinc on palm tree leaves. The value of k_d , which is determined from the slope of the plot, is found to be 0.21 mmol/(g · h^{0.5}) which indicates rapid diffusion and thus limited intra-particle diffusion contribution (4). This supposition is justified and supported by the observed rapid biosorption trend of zinc on palm tree leaves where 90% of zinc was removed within the first ten minutes.

Thermodynamics Analysis

Thermodynamics analysis in sorption studies usually involves studying sorption equilibrium at different temperatures. The main driving force in adsorption processes is the change in Gibbs free energy, ΔG^0 , which can be calculated using the equation

$$\Delta G^0 = -RT \ln K \quad (12)$$

where K is the distribution coefficient, which can be replaced by the Langmuir constant, K_L . Table 4 shows the values of K and ΔG^0 at different temperatures. As can be depicted from Table 4, the values of ΔG^0 were negative which confirms the feasibility of the biosorption of zinc on palm tree leaves and the spontaneous nature of the biosorption process. Moreover, the effect of temperature on adsorption equilibrium can be analyzed using the von't Hoff equation

$$\ln K = \frac{-\Delta H^0}{RT} + \frac{\Delta S^0}{R} \quad (13)$$

where ΔH^0 and ΔS^0 are the enthalpy and entropy changes, respectively. Figure 7 shows a plot of $\ln K$ against $1/T$. The slope and the intercept of this plot can be used to calculate ΔH^0 and ΔS^0 , respectively. The standard enthalpy and entropy changes of biosorption determined from Fig. 7 were 16.65 kJ mol⁻¹ and 0.067 kJ mol⁻¹ K⁻¹, respectively. The positive value of ΔH^0 indicates the endothermic nature of biosorption and the positive value of ΔS^0 confirms the increased randomness at the solid–solution interface

Table 4. ΔG^0 values for the biosorption of zinc on palm tree leaves at different temperatures

T (°C)	$-\Delta G^0$ (J/mol)
25.0	3184.7
30.0	3670.5
35.0	3879.8
40.0	4224.1

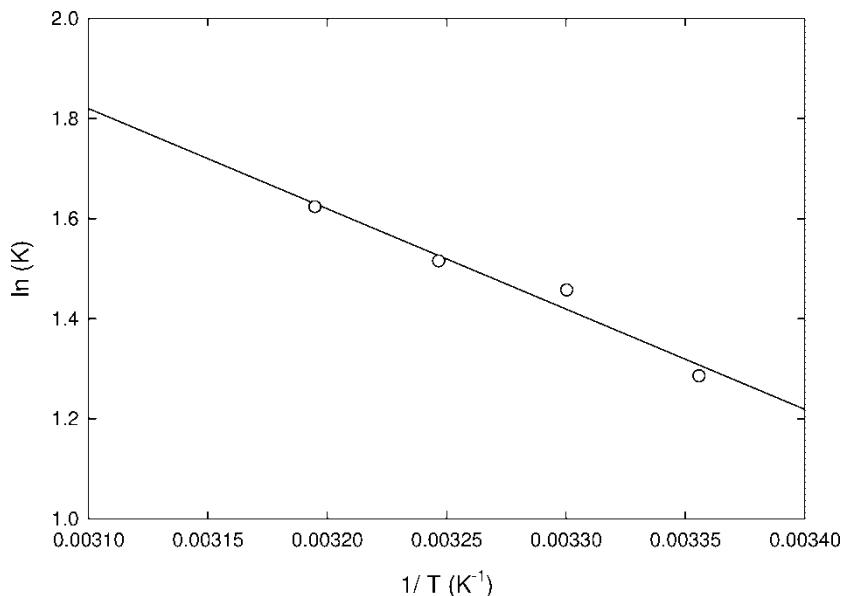


Figure 7. Application of von't Hoff equation for the biosorption of zinc on palm tree leaves (pH = 5.5 ppm, concentration of palm tree leaves = 2.0 mg/ml)

during the biosorption process which reflects the affinity of palm tree leaves for zinc.

Competitive Biosorption

In general, contaminated water contains more than single metal or pollutants other than heavy metals (35–37). Ethylenediamine tetraacetic acid (EDTA) is one of the common pollutants present in the wastewaters of many industries, such as metal plating, nuclear, pharmaceutical, pulp, and paper processing, and textiles (35). The presence of EDTA in these wastewaters is expected to cause interactive effects depending on many reasons, such as the initial metal concentrations, the initial concentration of EDTA, and the nature and dose of the biosorbent (35). The results of the biosorption of the binary $Zn^{2+} + EDTA$ are shown in Fig. 8. Figure 8 shows that the presence of EDTA resulted in suppression in zinc uptake. Further, the extent of suppression in zinc biosorption was also enhanced by increasing the EDTA concentration. The decreased zinc uptake in competitive conditions is thought to be a response to increased competition between like charged species for binding sites of the palm tree leaves. This progressive suppression in biosorption by EDTA indicates a degree of overlap in the biosorption site function at higher equilibrium concentration.

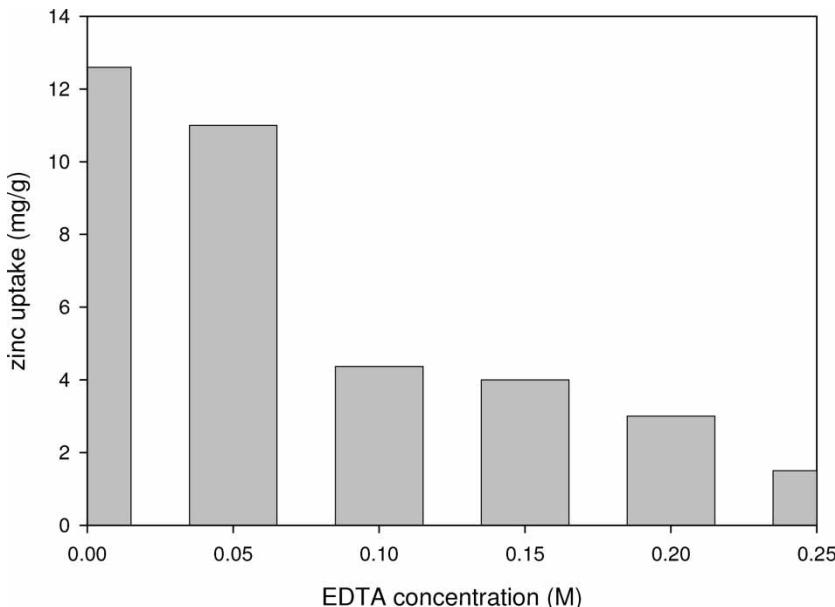


Figure 8. Effect of EDTA on the biosorption of zinc on palm tree leaves (Initial zinc ions concentration = 100 ppm, $T = 25^\circ\text{C}$, $\text{pH} = 5.5$, concentration of palm tree leaves = 2.0 mg/ml).

CONCLUSIONS

This study proved the practical feasibility of using palm tree leaves as a biosorbent for the removal of zinc ions from wastewaters. Biosorption of zinc by palm tree leaves was affected by temperature, biosorbent dose, and equilibrium pH. Thermodynamics studies confirmed the feasibility of the biosorption of zinc on palm tree leaves and the spontaneous nature of the biosorption process. The presence of EDTA was found to suppress zinc removal. The biosorption of zinc on palm tree leaves was found to follow pseudo-second order. The biosorption of zinc on palm tree leaves has been found to follow the Langmuir, Freundlich, Sips, and Gin isotherm models.

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