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Biosorption isotherm and kinetic studies for the removal of Pb (II) and Fe (II) ions from synthetic waste water using unmodified *Dennettia tripetala*

Tatah Verwiyeh Silas * and Asuelimen Steve Osagie

Department of Biochemistry, Federal University Wukari, Taraba State, Nigeria.

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Abstract

Various heavy metals have been released into the waters, causing serious pollution of water resources and endangering human health. Therefore, it is very important to study the removal of heavy metals from water. In this work, the adsorption of heavy metal ions including Pb(II), Cu(II), and Fe(II), onto *Dennettia tripetala* from aqueous solutions was investigated in mixed systems. The batch adsorption experiment for heavy metal ions on *Dennettia tripetala* mixed system was studied at different conditions including contact time, initial concentration, initial pH and adsorbent dosage. The results indicated that the adsorption kinetics followed the pseudo-first order model base on R² for Fe(II) which is 0.907, and pseudo-second order for Pb(II) with R² = 0.70 respectively. Hence the freundlich isotherm model is the best fit for Fe(II) with R² = 0.979 and for the Pb(II), it fits Temkin model with R² = 0.930. The final removal efficiencies and maximum adsorption capacity followed the order of Pb(II) > Cu(II) > Fe(II) at room temperature. The bio-sorption of heavy metals determined by the pH of the solution and the adsorbent dose, was found that the initial pH of 7 was optimal for the removal of Pb(II) and Fe(II), which was different from the optimum initial pH below or above 7. The results demonstrated that the electrostatic interaction between the surface of the *Dennettia tripetala* and heavy metal ions played an important role in the adsorption of Cu(II), Pb(II) and Fe(II) was promoted since none of the metal co-exist with one another.

Keywords: Dennettia tripetala; Heavy metals; Biosorption; Isotherm; Waste water

1. Introduction

Metals are substances with high electrical conductivity, malleability, and luster, which voluntarily lose their electrons to form cations. They are found naturally in the earth's crust and their compositions vary among different localities, resulting in spatial variations of surrounding concentrations [1]. The metal distribution in the atmosphere is monitored by the properties of the given metal and by various environmental factors [2]. Heavy metals by its nature have two most important characteristics, namely toxicity and persistency; therefore, disposal of waste containing these metals into receiving water bodies can be hazardous to both human and aquatic life [3]. Reports have it that the presence of heavy metals in water pose serious problems, because they may be mutagenic and carcinogenic [4]. Moreover, they can also cause severe damage to human beings such as dysfunction of kidneys, reproductive system, brain and central nervous system. Drinking water is mainly contaminated by metal ions from different sources especially wastewater and this is a serious environmental problem [5]. Heavy metals are significant environmental pollutants and their toxicity is a problem of increasing significance for ecological, evolutionary, nutritional and environmental reasons [6]. The most commonly found heavy metals in waste water include arsenic, cadmium, chromium, copper, lead, nickel, and zinc, all of which cause risks for human health and the environment [7]. Heavy metals enter the surroundings by natural means and through human activities. Various sources of heavy metals include soil erosion, natural weathering of the earth's

^{*} Corresponding author: Tatah Verwiyeh Silas

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crust, mining, industrial effluents, urban runoff, sewage discharge, insect or disease control agents applied to crops, and many others [1].

Although these metals have crucial biological functions in plants and animals, sometimes their chemical coordination and oxidation-reduction properties have given them an additional benefit so that they can escape control mechanisms such as homeostasis, transport, compartmentalization and binding to required cell constituents. These metals bind with protein sites which are not made for them by displacing original metals from their natural binding sites causing malfunctioning of cells and ultimately toxicity [1]. Previous research has found that oxidative deterioration of biological macromolecules is primarily due to binding of heavy metals to the DNA and nuclear proteins [8].

There are increasing concerns around the world about the discharge of heavy metals from industrial activities in to the environment. These contaminants are not degradable so linger in the soil and environment for a long time [9]. Industrial wastewaters are discharged directly or indirectly into the environment and the pollutants and its content are discharge into primary water resources through surface run-off (WHO, 1971). Consuming these undesired elements can cause different levels of toxicity in humans and animals even at low concentration [10]. Prolong exposure to lead(II) has been shown to cause various life-threatening conditions such as several forms of cancer, kidney damage, central nerve system damage, brain damage, dermatitis, dizziness, and liver problems [11] Lead has a half-life of about 600-3000 days and most of the inhaled Pb is accumulated in the bones.

The global need for a pollution free environment has continued to be abortive, due to incessant release of toxic heavy metal ions from industrial effluents that are detrimental to both flora and fauna. They are high priority pollutants because of their relatively high toxic and persistent nature in the environment. These metals in the form of inorganic compounds from natural and anthropogenic sources continuously enter the aquatic ecosystem where they could pose serious threat as they are passed along the food chain. Therefore, there is a need for further remediation.

Using chemically treated adsorbents may create a need for further remediation procedures to clean the water posttreatment, but using agricultural waste materials as adsorbents reduces the possibility of releasing unwanted byproducts into the water. The use of agricultural waste materials such as *Dennettia tripetala* to treat contaminated water is a low cost and effective way to deal with the two areas (water treatment and waste management).

2. Material and methods

2.1. Preparation of adsorbent

Dennettia tripetala roots were obtained within Wukari Local Government area of Taraba State, Nigeria as inexpensive, natural and safe adsorbent. It was thoroughly washed with deionized water in order to remove dust and other debris. They were then oven dried for 24 h at 23°C for the reduction in moisture content. The dried biomass was ground and sieved to particles (250 μ m). The sieved powder was stored and used for batch experiments.

2.2. Preparation of stock solution

The absorbate solution was prepared through a serial dilution procedure using a high purity stock standard lead and copper (1000 mg/L) solution.

Therefore, $1.57 \text{ g Pb}(C_2H_3O_2)_2$ and 2.9 g of FeCl_3 the stock solution was then transferred to 1000 ml volumetric flask The prepared adsorbate was diluted to the required different concentrations before starting the experiment.

2.3. Bach adsorption

Batch adsorption experiments were conducted using lead ion solutions prepared by diluting 1000 ppm of the stock lead ion solution. All experiments were conducted by adding a known amount of the adsorbent to 100-ml volume reagent bottles. After adding known amount of adsorbent, the mixtures in the bottles were agitated for a predetermined time at room temperature using a reciprocating shaker.

2.4. The effect of time

The lead and iron adsorption efficiency from aqueous solutions with an initial concentration of 50 ppm on 5 g/ 60 ml of *Dennettia tripetala* was observed depending on contact time of 0, 20, 40, 60, 80 and 120 min. The adsorption efficiency qe is calculated depending on the metal concentration of 50 ppm before and after attaining equilibrium state in the aqueous solution.

The Equilibrium contact times were determined for each adsorbent after testing different predetermined time intervals. The adsorption capacity was calculated using the equation:

$$qe = \frac{(CO - Ce)V}{W}$$
....(1)

where qe is the equilibrium uptake (mg/g), C0 the initial metal ion concentration (mg/l), Ce the equilibrium metal ion concentration (mg/l), V the volume of the solution (l) and w the mass of adsorbent (g). Equilibrium isotherms were obtained by conducting the adsorption experiments with initial lead ion concentrations. Kinetic studies were conducted using batch adsorption experiments for different time lengths with an initial lead ion concentration of 50 ml.

2.5. Effect of PH

The pH of an aqueous solution is described as a significant parameter that influences the adsorption process. During this process, the functional groups, surface charges, degree of ionization and solubility of the adsorbent are frequently responsible for the binding of metal ions onto the adsorbent.

The percentage of lead removal was calculated using:

Pb removal (%) =
$$=\frac{(Ci - Ce) \times 100}{Ci}$$
.....(2)

2.6. Effect of adsorbent dose

The heavy metal adsorption is observed depending on the effect of adsorbent dose at 60 min contact time for an initial 50 mg/l heavy metal concentration under 230C. The samples were taken at the different adsorbent dose and analyzed for their lead ion concentration.

2.7. Effect of Initial Metal Ion Concentration

The amount of metal ions sorbed is a function of the initial concentration of the metal ion, making it an important factor in effective adsorption. The sorption capacity of *Dennettia tripetala* for Fe (II) and Pb(II) ions for different initial concentration of 10, 20, 40, 60, and 80mg/l was investigated.

2.8. Adsorption of multi-component heavy metal ions

This analysis was done in order to obtain a deeper understanding of the adsorption efficiency and competition between heavy metal ions, adsorption experiment was performed in deionised water of multicomponent mixed system containing Pb(II), Cu(II), Fe(II) of different concentration.

After such combination, the solution was filtered and the aqueous phase of every sample was analyzed for its Pb ions concentration using Atomic Adsorption Spectrophotometer (AAS).

2.9. Equilibrium isotherm modelling

The knowledge of sorbate/sorbent interaction at equilibrium is an essential tool in adsorption design and the use of equilibrium isotherm is one of the common methods deployed to obtain this information. According to Hutson et al. [12], the essential issue is the understanding of the specific relationship between the pollutant concentration and its uptake degree by the solid phase at constant temperature and this is used to construct adsorption isotherms. Equilibrium studies were carried out for the removal of Lead (II) and iron (II) ions from aqueous system using the *Dennettia tripetala*.

The pseudo-first-order and pseudo-second-order adsorption kinetics based on equilibrium adsorption are represented as follows:

$$ln (qe - qt) = lnqt - k1t t/qt = 1/k q 2 + 1/qe t$$

where qe and qt are the amounts of metal ions adsorbed onto the *Dennettia tripetala* hust (mg/g) at equilibrium and at a time, t, respectively. k1 and k2 are the rate constants for pseudo-first-order and pseudo-second-order kinetics, respectively. The first-order constant (min-1) was determined in linear form by plotting ln(qe - qt) against t. A plot of t qt against t was used to determine pseudo- second-order constant (mg/g min).

The Langmuir and Freundlich isotherms were used to characterize the adsorption process. The Langmuir, Freundlich and Tisotherm models were evaluated using the non- linear method, a trial and error procedure. This approach is designed to obtain isotherm parameters from the models by minimizing the respective coefficient of determination between experimental data and isotherm models [13]. To determine the goodness of fit of the isotherm models to the experimental data using nonlinear regression, the optimization procedure requires that error functions be defined to enable the fitting of the model parameters with the experimental values, the coefficient of determination (r2) were used as error parameter for each model.

2.10. Kinetic model

The adsorption kinetics and rate constants were determined from kinetic models including the pseudo-first-order and pseudo-second-order models. Adsorption kinetics provides information on the reaction pathways and the mechanisms of the adsorption of sorbate by the sorbent.

The pseudo-first-order and pseudo-second-order adsorption kinetics based on equilibrium adsorption are represented as follows:

$$\ln (qe - qt) = \ln qt - k1t t/qt = 1/k q 2 + 1/qe t$$

where qe and qt are the amounts of metal ions adsorbed onto the *Dennettia tripetala* hust (mg/g)at equilibrium and at a time, t, respectively. k1 and k2 are the rate constants for pseudo-f irst-order and pseudo-second-order kinetics, respectively. The first-order constant (min-1) was determined in linear form by plotting ln(qe - qt) against t. A plot of t qt against t was used to determine pseudo- second-order constant (mg/g) min).

2.11. Data Analysis

The experimental equilibrium and kinetic data for the sorption of Pb(II) ions onto the *Dennettia tripetala* adsorbent were modelled using the equilibrium and kinetic models described in this study. To determine the model which best describes the sorption a number of fitting parameters were used to correlate the experimental data based on the magnitude of the correlation coefficient for the regression, that is the model which gives an R2 value closest to unity is deemed the best fit [14].

3. Results and discussion

3.1. Effect of contact time



Figure 1 Effect of time on the adsorption of metal ions

Removal of metal ions increases as the time increases and achieves a maximum at time 120 min, as shown in Figure 4.1 indicates that lead and iron concentration in the aqueous solution decreases promptly for the initial 20 min and virtual completion of removal process within 120 min which was 98%. The process of removing metal ion exhibits in two

stages: The first stage involves the high removal rate whereas the second stage of adsorption process determines the equilibrium time (i.e.) contact time of unchanging Pb and Fe concentration in the aqueous solution after 20 min, which can be easily described by the interaction of the Pb and Fe ions at the vacant initial stage [15]. Initially high adsorption rate exhibits because of the presence of active adsorbing site on adsorbent surface, which is indicated by the larger uncovered surfaces offered by increased amount of *Dennattia tripetala*. In addition to this, the driving force of adsorption is defined as the difference of Pb and Fe ion concentration and the exerted solid/liquid interfaces. High value of driving force results in fast adsorption. In the curve, low slopes indicate the slow second stage because of the low diffusion velocity of ions in the pores of *Dennattia tripetala*. In case of initial solution with lower metal ion concentration (50 mg/l), better adsorption capacity is attained.

3.2. Effect of PH

In Fig. 2, the optimum pH values ranged from 4, 6, 7, 8 and 10 for the studied metal ions. It was observed that maximum removal of metal ions occurred at pH 7 and a further increase in pH above their optimum caused the formation of precipitates. At lower pH, the adsorbent is protonated and the protons compete with the metal ions, resulting in less adsorption. The interaction between these metal ions increased with pH, and at higher pH, the presence of negatively charged functional groups such as hydroxyl and oxalate ions ensure the metal oxalate complex formation, thus influencing the adsorption of metal ions onto the adsorbent. The electrostatic interaction between the positively charged metal ions and the negatively charged oxalate ions, a bidentate ligand, is responsible for the formation of the metal oxalate complexes.

A rare interpretation of the effect of pH on the adsorption process via sorption edge, in which the concentration of the adsorbate is kept constant while the pH is varied, the maximum percentage sorption was attained at optimum pH.7 as shown in Fig. 2.



Figure 2 Effect of pH

3.3. Effect of adsorbent dose

Figure 4.3 dictates the increase of Pb(II) and Fe (II) ion removal percentage with increase in adsorbent. Increase in adsorption for Pb ions from 94 to 96% and for Fe ions from 94 to 99% exists as the adsorbent dose from 2 to 10 g/ 60 ml increases after 60 min.

The number of adsorption site or surface area tends to increase based on the adsorbent weight thus resulting in higher percentage removal of metal ions at high dose [16]. Thus the adsorbed metal ions expressed in per unit weight of adsorbent (q) decreases with decrease adsorbent dose as revealed in Figures 3. This mainly occurs because of the lower concentration of metal ions at high adsorbent dose thus enabling the system to attain equilibrium state faster than other parameters.



Figure 3 Effect of adsorbent dose

3.4. Effect of multi-component metal ions

Effect of multicomponent mixed system containing Pb(II), Cu(II), Fe(II) of different concentration, the percentage removal increases in order of: Pb(II)>Cu(II)>Fe(II) as shown in Figure 4, It can be seen that the removal rate of Pb(II) was higher than the rest of the metal ions under the condition of 5 dosages of initial concentration of 15 mg/L and shaking time of 60 min at 285 rpm. Hence it has be proved that Pb(II) could not coexist with other three heavy metal ions in this system.



Figure 4 Effect of multi-component heavy metal ion

3.5. Effect of initial metal ion concentration

The sorption capacity of *Dennettia tripetala* for Fe(II) and Pb(II) ions as shown in Figure 5 increased with increasing metal ion concentration from 10 to 80mg/l, resulting to an increase metal ion concentration gradient that overcomes the resistance to mass transfer of metal ions between the aqueous phase and the adsorbent. A higher concentration in a solution implies a higher concentration of metal ion to be fixed on the surface of the adsorbent [16]. The high sorption capacity of *Dennettia tripetala* for Pd(II) and Cu(II) ions as shown in figure 4.5 is possibly attributed to its surface porosity, better still may be due to its larger surface area and good cation exchange capacity. Dawodu et al. [17] reported a similar observation on simultaneous adsorption of Ni(II) and Mn(II) ions from aqueous solution onto a Nigerian

kaolinite clay. A higher metal concentration saturated the adsorbent sites more quickly thereby decreasing the overall percentage metal removal.



Figure 5 Effect of initial metals ions concentration

3.6. Equilibrium Isotherm models

From the study, metal ion loading on the adsorbent was calculated and in this study, the Langmuir and Freundlich isotherms were used to characterize the adsorption process. The Langmuir and Freundlich isotherm models were evaluated using the non- linear method, a trial and error procedure. This approach is designed to obtain isotherm parameters from the models by minimizing the respective coefficient of determination between experimental data and isotherm models [18]. To determine the goodness of fit of the isotherm models to the experimental data using nonlinear regression, the optimization procedure requires that error functions be defined to enable the fitting of the model parameters with the experimental values, the coefficient of determination (r2) were used as error parameter for each model.

3.7. Langmuir isotherm

The Langmuir adsorption isotherm assumes that monolayer adsorption exists at all surface sites that are homogeneity, with the ability of no interaction of adsorbed molecules with the neighbouring adsorption sites. The linear Langmuir equation is represented as follows:

where Ce is the equilibrium concentration (mg/dm3), qe is the amount (mg/g) adsorbed at equilibrium time, and Qm and b are Langmuir constants related to maximum adsorption capacity (mg/g) and energy of adsorption related to the heat of adsorption (L/mg), respectively. The Langmuir isotherm parameters were used to calculate the affinity between the adsorbent and adsorbate via dimensionless separation factor, RL, as determined using the following equation:

where b is the Langmuir constant and Co is the initial concentration of metal ions. The RL values promulgate whether the adsorption is irreversible (RL = 0), favourable (0 < RL < 1) or linear if (RL =1) or unfavourable if (RL > 1). Table 1 shows low correlation coefficients (R2) of 0.712 for

Pb(II) and 0.673 for Cu(II), compare to freundlich model, indicating that the adsorption does not followed the Langmuir isotherm closely.

3.8. Freundlich isotherm

The empirical Freundlich isotherm model is applied to describe the adsorption on a non-uniform (heterogeneous) surface with the interaction between adsorbed molecules in the reversible and non-ideal adsorption process. The Freundlich adsorption isotherm linear form is given as:

where KF and n are Freundlich constants representing coefficient and intensity, respectively. The experimental values for n and KF based on the slope and the intercept, respectively. As can be seen in Table 5, the R2 values for both Pb(II) and Fe(II) ions, 0.979 and 0.920 respectively were higher than those of the Langmuir model, indicating a good fit of the Freundlich model to the adsorption process. Though the values of n obtained for both metal ions lie between 1 and 10, suggesting favourable adsorption.

Table 1 Equilibrium Isotherm Parameters for the Biosorption Process

Isotherm Models	Cu (II)	Pb (II
Langmuir model		
qL (mg/g)	6.711	6.511
KL (L/mg)	0.016	0.031
R ²	0.712	0.673
Freundlich model		
KF (L/g)	0.459	0.668
1/n	0.741	0.601
R ²	0.979	0.920
Temkin model		
A (L/g)	5.781	1.652
B (mg/g)	2.245	1.770
R ²	0.973	0.930

3.9. Kinetic model

Table 2 describes the kinetics of the adsorptions of studied metal ions on by using pseudo-first and pseudo-second-order models.

For the pseudo-first-order model, the low k1 suggests that a slow adsorption process takes place, while for the pseudosecond order model, k2 values were high indicating an increase in adsorption rates. The R² values for the adsorption kinetics of the metal ions were higher for the pseudo- second-order model compared to those of the pseudo-first-order model. Thus, it appears that pseudo-second-order model is a better model fitting the kinetics of the adsorption of the metal ions employed in this study.

3.9.1. Pseudo-first order

The model presented a good fit to the kinetic data base on r2 values obtained for the Cu(II) ions as shown in table 2. However, the values of the calculated sorption capacity (qecal) were higher than those of experimental sorption capacity (qexp) obtained, indicating a disagreement.

3.9.2. Pseudo-second order

The values of qecal for pseudo-second order were almost equivalent to the experimental values (qe exp) obtained for lead (II), confirming it is the model that best described the kinetic mechanism of the removal of (Pb) ions from wastewater in this study.

Kinetic models	Cu (II)	Pb(II)	
qe exp (mg/g)	0.570	0.391	
Pseudo-first order			
q _e cal, (mg/g)	0.604	1.481	
K1, (min-1)	0.085	0.093	
R ²	0.908	0.701	
Pseudo-second order			
q _e cal, (mg/g)	7.209	0.401	
K2 (gmg-1min-1)	0.068	0.013	
h (mg/gmin)	1.292	0.354	
R ²	0.581	0.970	

Table 2 Kinetic parameters for the adsorption of Cu (II) and Pb(II)

4. Conclusion

From the experimental investigation, heavy metal biosorption using inexpensive and efficient biosorption from agricultural waste materials has showed a promising replacement for existing conventional system. From the listed results, it can be concluded that *Dennattia tripetala* is a good adsorbent of lead (II) and copper (II) ions from solutions and invariably from wastewater. The kinetic studies show that the biosorption process followed the pseudo second order kinetic model. Freundlich model showed the best fit for the experimental data. Hence in comparison with other biosorbent from literature, it is can compete favorably with other available synthetic and natural adsorbents.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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