



(RESEARCH ARTICLE)



Biosorption of Cu (II) and Pb (II) ions using acid modified *Senna tora* pods with a focus on isotherms and kinetics studies

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Abstract

The removal of heavy metals by biosorption method using agricultural waste and plant materials has become a powerful technology that is been explored for the treatment of domestic and industrial waste water due to its easy operating requirements and low cost. This work reports the application of *Senna tora* pods for the elimination of heavy metals (Cu and Pb ions) by biosorption in batch system. The effects of pH, contact time, dosage and initial concentration were evaluated. The kinetic and isotherms models for the Cu(II)/Pb(II) biosorption were tested basically by the pseudo-first-order and pseudo-second order kinetic models. Langmuir, Freundlich and Temkin isotherms models have also been used to fit equilibrium adsorption data. The monolayer sorption capacity (q_L) of Cu(II) was found to be 0.791 mg/g while that of Pb(II) was 0.51 mg/g. The low Langmuir constant (K_L) values suggests that the adsorption process was largely due to physisorption mechanism. The adsorption kinetics follow a pseudo-first-order model for Cu(II) and pseudo-second-order model for Pb(II), and the equilibrium data is suitably fitted by the Langmuir models than the Freundlich due to the high R^2 values of 0.989 and 0.97 for Cu(II) and Pb(II) ions respectively. Consequently, there exists a closeness of the calculated sorption capacity (q_{cal}) to the experimental sorption capacity (q_{ex}). In conclusion, the study generally showed that *Senna tora* pods is a potential and efficient adsorbent that could be explore for the bioremediation of heavy metal contaminated industrial wastewater

Keywords: Bioremediation; Adsorption; Wastewater; Heavy metals; Isotherms

1. Introduction

Industrial activities in developed, underdeveloped and developing countries in recent times have faced a lot of problems due to improper disposal of wastes [1]. These have resulted in emerging contaminants in water bodies causing severe health implications [2]. Effluents from industries such as tanning, mining, textile, fertilizer, pharmaceutical and inputs from other anthropogenic activities have contributed to the increase in contaminated waters all over the globe [3]. Therefore, it is very important to do check and balancing of water quality, especially when their heavy metal ion concentrations are higher than the permissible limits.

The world's main water resources are being polluted by the undesirable organic and inorganic waste released into the environment because of the advance of civilization and rapid industrialization. A large proportion of water pollutants that affect our environment results from industrial activities and their ever increasing nature have led to the present state of our global environment [4]. This is because of the huge chemical input associated with these processes and the inefficient resource utilization of the input components leads to huge generation of waste streams (The low efficiency of the treatment processes used in these industrial sites for the treatment of industrial effluents further increases the pollutant load in treated discharged effluents. The presence of toxic contaminants such as heavy metals can damage the ecosystem and the human body [5]. Many studies have shown that these metals are toxic even at low concentrations.

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This could, in turn, cause accumulative poisoning, cancer and brain damage when found above the tolerance level [6]. Copper is one of the toxic heavy metals which are common pollutants of the environment.

Senna tora (originally described by Linnaeus as *Cassia tora*) is a plant species in the family Fabaceae and the subfamily *Caesalpinioideae* [7]. Its name is derived from its Sinhala name tora. It grows wild in most of the tropics and is considered a weed in many places. The plant is abundant in Africa especially in Nigeria. The plant can grow 30-90 centimeters (12-35inc) tall and consists of alternative pinnate leaves with leaflets mostly with three opposite pairs that are obovate in shape with a rounded tip. During October to February, *senna tora* grows in dry soil from sea level upto 1800 metres. The seed can remain viable for up to twenty years. Up to 1000 plants can emerge per square meter following rain.

Senna tora has an external germicide and antiparasitic properties and has been used for treating skin diseases such as leprosy, ringworm, itching and psoriasis and also for snakebites [8]. Other medicinal provisions from plant parts include balm for arthritis using the leaves. The plant has a high binding affinity to heavy metals, therefore are used in biosorption study of heavy metals from synthetic waste water [9].

Several analytical methods have been carried out for the removal of heavy metals from synthetic waste water this includes the chemical precipitation methods, ion exchange method, ultra-filtration method, reverse osmosis and electro dialysis [10]. But these methods were reported to be very expensive. The high cost of chemicals in the market has motivated me to use low cost biomass as biosorbent for the removal of heavy metals from waste water.

A lot of agricultural waste materials are disposed in our environment; these agricultural wastes have binding affinity to heavy metals which can be used for the biosorption study [11]. The removal of this heavy metal by biosorption method using agricultural waste has been become a powerful technology that is been explored for the treatment of domestic and industrial waste water due to its easy operating requirements and low cost. This research is aimed at investigating the biosorption of Cu(II) and Pb(II) ions using acid modified *Senna tora* pods with a focus on isotherms and kinetics studies.

2. Material and methods

2.1. Preparation of adsorbent

Sena tora pods were obtained within Wukari Local Government Area of Taraba state of Nigeria, it was thoroughly washed and rinsed with distilled water to ensure the removal of dust and any soluble impurities. It was then kept at room temperature and expose to air for natural drying for 24hrs. The sample was then placed in oven at 110°C overnight, to remove moisture and any other volatile impurities that may exist.

The dried leaves were later ground into fine powder, 1.0 M HCl acid was dissolved in water, the solution was then added to the biosobent for few minutes after which it was filtered. The treated pods were air dried and stored in desiccator to prevent moisture and being ready for batch experiment.

2.2. Stock solution preparation

Analytical grade chemicals were used in preparing all the solutions that were used in the study. Synthetic stock solution of Cu(II) and Pb(II) effluent (1000 mg/l) were prepared by dissolving separate 1.76g Cu(II) and 1.57g Pb(II) in 1000 ml of deionized water. All other concentrations of the metal ions were prepared by serial dilution of the stock solutions [12]. Hydrochloric acid 0.1 M and 0.1 M NaOH was used for adjustment of the aqueous solution pH.

2.3. Adsorption experiments

Batch adsorption techniques were carried out by varying contact time, solution pH, initial concentration, adsorbent load and temperature on the dried *Senna tora* powder. Adsorption experiments were carried out by adding accurate amounts of the adsorbent to 60 cm³ volume of the wastewater in 100 ml reagent bottle placed on an orbital shaker in order to attain the equilibrium time. The solution pH (4,6,7,8 and 12), adsorbent dosage (2,4,6,8 and 10 g), initial metal ion concentrations (10, 20, 40, 60 and 80 mg/L) were varied to study their effects on the metal ion removal efficiency. The analysis of the metal ion concentrations using atomic adsorption spectrophotometer (AAS) was done after filtering with Whatman 42 filter paper. The metal uptake and the percentage removal were calculated according to the following respective equations:

$$q_e = \frac{(C_0 - C_e)V}{w} \dots\dots\dots (1)$$

$$\% \text{ Removal} = \frac{C_0 - C_e}{C_0} \times 100 \dots\dots\dots(2)$$

where q_e is the equilibrium uptake (mg/g), C_0 the initial metal ion concentration (mg/l), C_e 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 metal ion concentrations. Kinetic studies were conducted using batch adsorption experiments for different time lengths with an initial lead ion concentration of 50 ml. In these experiments, samples were taken at the different time periods and analyzed for their metals ion concentration.

2.4. Analytical methods

Identically, prior to measurement, all samples were separated from supernatant after adsorption, filtered through whatman filter paper (0.45 μm pore size) The residual heavy metal concentrations of all samples were measured using an atomic adsorption spectrophotometer (AAS). All adsorption experiments were performed in replicate with a relative standard deviation of less than 1%.

2.5. Data Analysis

The experimental equilibrium and kinetic data for the sorption of Cu(II) and Pb(II) ions onto the *Senna tora* 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 R^2 value closest to unity is deemed the best fit [12].

3. Results and discussion

3.1. Effect of pH

The pH has a great influence on the M(II) removal by adsorption, because it directly affects the surface charge and the nature of ionic species of the adsorbates. In this context, the pH effect was studied in the range (4-10). The removal capacity of Cu(II)/Pb(II) increases with increasing pH (Figure 1); the minimal adsorption at pH 4 is due to the high concentration of H^+ ions. At low pH, higher concentration and mobility of H^+ ions favour H^+ sorption compared to metal ions, this creates a competition between the protons and metal ions for the binding sites of the adsorbent. According to Onundi et al. [13] metal ions are more soluble in solution at lower pH values and this reduces their sorption. However, when the pH increases, the biomass surface becomes negatively charged, leading to increased Cu(II)/Pb(II) uptake and reaches equilibrium at pH 7. The retention capacity of M(II) decreases above pH 7 because of the apparition of insoluble $\text{M}(\text{OH})_2$, and all hydroxides have a solubility product smaller than 10^{-15} [14]. Similar tendencies were found in adsorption processes using diverse agricultural waste biomass (AWBs). Giri et al. [15] reported a similar trend on studies of the effect of pH on the removal of Cr (VI) using *Eichhornia crassipes* root activated carbon. However, at higher pH metal ions tend to precipitate out of solution. Therefore the removal of metal ions at higher pH values is due to the formation of metal ion precipitates rather than sorption [16, 17].

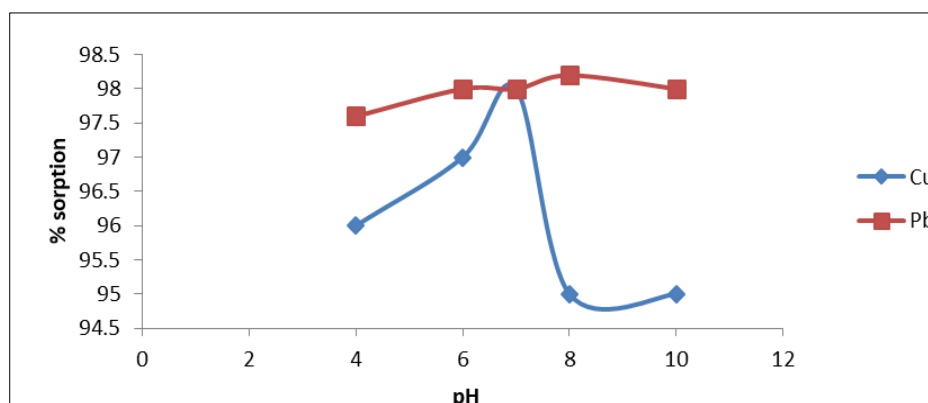


Figure 1 Effect of pH on sorption capacity of Cu and Pb onto *Senna tora* pods

3.2. Effect of contact time

The effect of contact time on the adsorption of Cu(II) and Pb(II) ions onto *Senna tora* pods is shown in Figure 2. An initial increase in percentage removal with increased contact time from 0-20 minutes was observed, and adsorption became fairly stable over time. Equilibrium removal was achieved around 40 min for Pb(II) and 80 min for Cu(II), after which further increases in contact time did not result in significant adsorption. Maximum adsorption was achieved at 120 min for both metal ions, and this time was used in all experiments to ensure maximum removal. The faster removal rate with Cu(II) than with Pb(II) may be due to the smaller ionic radius of Cu(II) than Pb(II), which makes for easier, more rapid diffusion to the surface of the adsorbent. The rapid adsorption during the initial stages might be due to the presence of abundant active sites on the surface of *Senna tora* pods, which become saturated with time. Initially, sorption is controlled mainly by diffusion from the bulk to the surface of the adsorbent, whereas, later, sorption is probably an attachment-controlled process due to the presence of fewer active sites [18, 19].

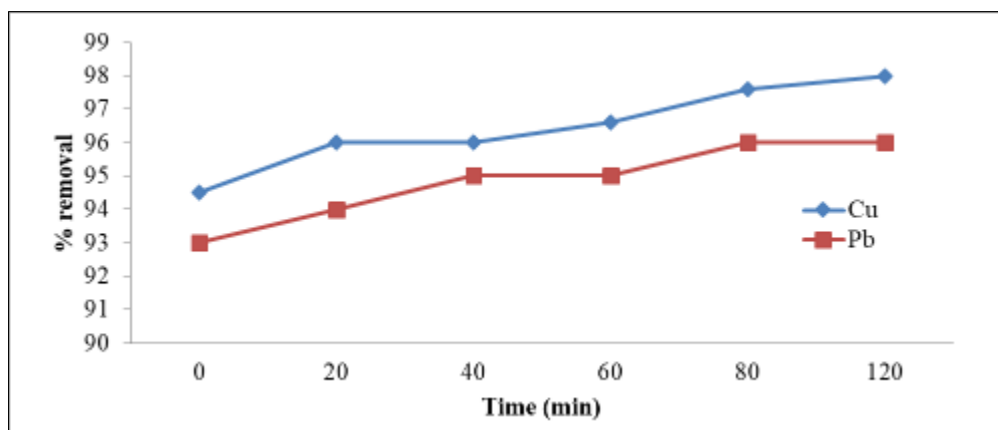


Figure 2 Effect of time on the sorption of Cu(II) and Pb(II) onto *Senna tora* pods

3.3. Effect of dosage

The greater biosorbent dose provides more active biosorption site throughout the biosorption reaction causing a reduction in equilibrium biosorption capacity per unit mass of biosorbent. Adsorbent dosage has proved to be a useful parameter for determining the capacity of an adsorbent for a given initial metal ion concentration. The percentage removal of Cu(II) and Pb(II) ions in this study, increased with increasing adsorbent dosage (Figure 3). Increasing the dose from 2 to 4 g led to minimal increase in percentage removal of Cu(II) from 95 to 96% while percentage removal of Pb(II) increased from 97.5 to 98%. This is due mainly to an increase in the number of available exchangeable binding sites for metal ion sorption [20, 21]. The equilibrium sorption capacity per unit mass of the adsorbents decreased considerably with increase in sorbent dose for both metal ions at 6 g. Conversely, a higher adsorbent dose may provide more active sorption sites, so that adsorption sites remain unsaturated during sorption, leading to their full utilization [22, 23]. The stability of M(II) uptake with increases in the biosorbent dose is due to the split in the flux between the M(II) concentration in the solution and the sorbent surface [14].

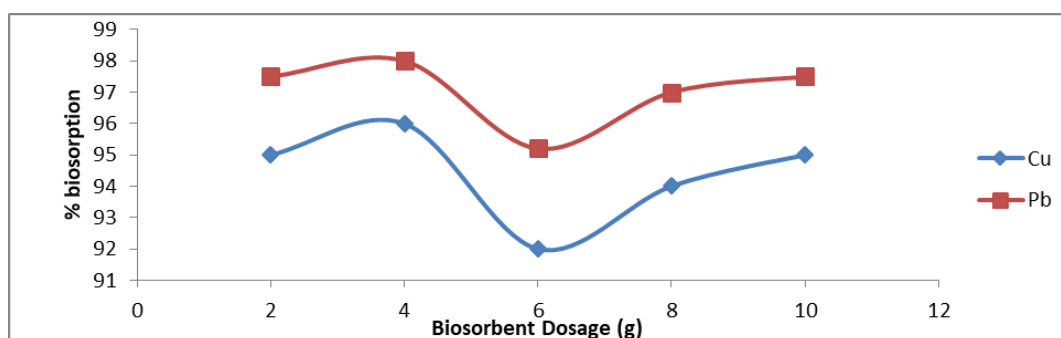


Figure 3 Effect of biosorbent dosage on sorption capacity of Cu and Pb onto *Senna tora* pods

3.4. Effect of initial concentration

At higher initial concentrations the active site of biosorbent is usually surrounded with more ions in the solution, hence the equilibrium biosorption capacity increases with increasing the metal ion concentration. The effect of the Cu(II) and Pb(II) initial concentrations on the biosorption by *Senna tora* pods is investigated by varying their initial concentrations in solution from 10 to 80 mg/L, maintaining the other parameters constant. The results show that the retention capacity of metal by the biomass increases with raising the ions concentrations in solution (Figure 4). This is due to the interaction between ions and biosorbent which provides the driving force to overcome the resistance to the mass transfer of Cu(II)/Pb(II) ions between the solution and solid phase. In addition, the enhanced biosorption with the Cu(II)/Pb(II) concentrations is due to an increase in the electrostatic interactions (physical adsorption versus covalent interactions) [24] The heavy metals absorption can be attributed to various mechanisms of ion exchange and adsorption processes [25, 26].

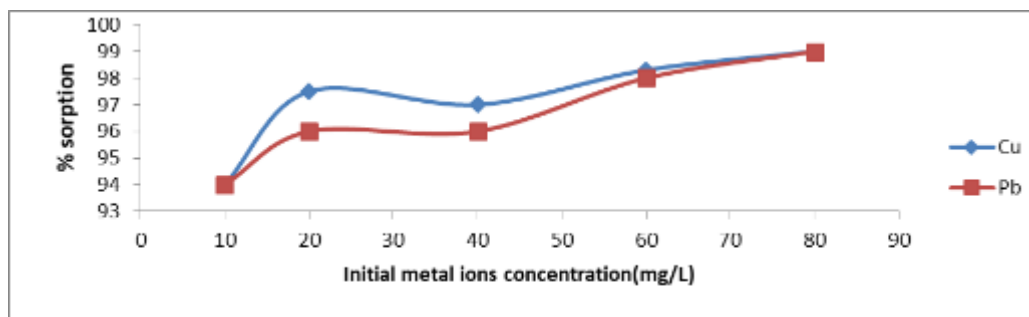


Figure 4 Effect of initial concentration on sorption capacity of Cu and Pb onto *Senna tora* pods

3.5. Biosorption of Multi-Component Heavy Metal Ions

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) and Fe(II) of different concentrations as shown in Figure 5. The evaluation of the effect of combining these different metals showed that they favorably compete for binding site, however, Cu(II) which has the strongest binding site has demonstrated the highest percentage sorption.

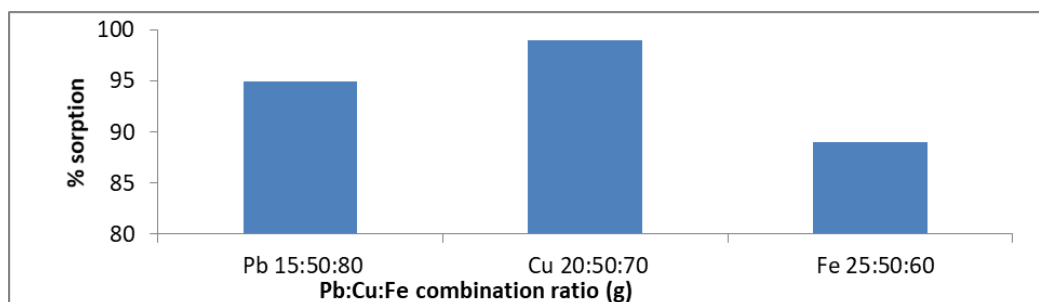


Figure 5 Biosorption of multi-component heavy metal ions by *Senna tora* pods

3.6. Biosorption Kinetic

Adsorption kinetics provides information on the reaction pathways and the mechanisms of the adsorption of sorbate by the sorbent. Table 1 describes the kinetics of the adsorptions of studied metal ions on powdered *Senna tora* by using pseudo-first and pseudo-second-order models. The biosorption kinetics of Cu(II) and Pb(II) on *Senna tora* pods were undertaken to establish the adsorption equilibrium time and kinetic parameters. The validity of each model was verified by the fitness of the correlation coefficient (R^2). The $q_e(\text{cal})$ values calculated from the pseudo-first-order model for Cu(II) is very close to the experimental one and the R^2 values converge to 1, indicating the validity of the pseudo-first order [27]. By contrast, in the pseudo-second-order model for Cu(II), the calculated $q_e(\text{cal})$ values differs significantly from $q_e(\text{exp})$. Whereas for Pb(II), the $q_e(\text{cal})$ values calculated for the pseudo-first-order model differs from the experimental one, but in the pseudo-second-order model, the calculated $q_e(\text{cal})$ values is very close to $q_e(\text{exp})$ and the R^2 values converge to 1, indicating the validity of the pseudo-first order [27].

Table 1 Kinetic parameters for biosorption of Cu (II) and Pb(II) by *Senna tora* pods

Kinetic models	Cu (II)	Pb(II)
q_e exp (mg/g)	0.45	1.2
Pseudo-first order		
q_{ecal} , (mg/g)	0.674	4.481
K_1 , (min ⁻¹)	0.045	0.033
R^2	0.983	0.82
Pseudo-second order		
q_{ecal} , (mg/g)	4.399	1.403
K_2 (gmg ⁻¹ min ⁻¹)	0.048	0.023
h (mg/gmin)	1.592	0.954
R^2	0.781	0.99

3.7. Equilibrium isotherm models

The most frequently used isotherms, the Langmuir, Freundlich, Temkin and Dubinin–Radushkevich models, were therefore used to analyse the data.

The Langmuir Isotherm model describes monolayer adsorption onto the surface of an adsorbent with a finite number of identical adsorption sites and no interaction between sites. The model is expressed as:

$$C_e/q_e = 1/q_L K_L + C_e/q_L \dots\dots\dots(3)$$

q_L (mg/g) is the monolayer adsorption capacity of the adsorbent, and K_L (L/mg) is the adsorption constant, which reflects the affinity between the adsorbent and adsorbate. q_L and K_L were determined from the slope and intercept of the plots of C_e/q_e versus C_e . An essential feature of the Langmuir isotherm can be expressed in terms of a dimensionless separation factor (R_L), defined as:

$$R_L = 1/[1 + K_L C_0] \dots\dots\dots(4)$$

Adsorption is said to be favourable if $0 < R_L < 1$ and unfavourable if $R_L > 1$. Table 2 shows high correlation coefficients (R^2) of 0.989 for Cu(II) and 0.970 for Pb(II), indicating that the adsorption followed the Langmuir isotherm closely.

The Freundlich Isotherm model is based on the assumption that sorption takes place on a heterogeneous adsorbent surface, where the sorption energy distribution decreases exponentially and can be expressed as:

$$\log q_e = \log K_F + [1/n] \log C_e \dots\dots\dots(5)$$

Where K_F (mg/g)(mg/L)^{1/n} and n are the constants for adsorption capacity and intensity, respectively. A plot of $\log q_e$ versus $\log C_e$ gives a straight line of slope $1/n$ and intercepts $\log K_F$. As seen in Table 2, the R^2 values for both Cu(II) and Pb(II) ions, 0.679 and 0.720 respectively were lower than those of the Langmuir model, indicating a poor 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. Hence equilibrium data is suitably fitted by the Langmuir models than the Freundlich model.

Table 2 Equilibrium Isotherm parameters for biosorption of Cu (II) and Pb(II) by *Senna tora* pods

Isotherm models	Cu (II)	Pb (II)
Langmuir model		
q _L (mg/g)	0.791	0.51
K _L (L/mg)	0.017	0.018
R ²	0.989	0.97
Freundlich model		
K _F (L/g)	0.489	0.638
1/n	0.646	0.401
R ²	0.679	0.72
Temkin model		
A (L/g)	2.781	1.652
B (mg/g)	2.245	1.77
R ²	0.773	0.73

4. Conclusion

This study has demonstrated that acid modified *Senna tora* powdered can be used as a natural, promising, economic and environmentally friendly adsorbent for Cu(II), Pb(II) and Cu(II) from synthetic waste water. The batch adsorption method is dependent on the contact time, dosage, solution pH, initial metal ion concentrations and temperature. The experimental data in the adsorption process indicated good correlations with the pseudo-second-order kinetic model and Langmuir isotherm. Based on this study, the readily available *Senna tora* plant which is nuisance to the environment could be used as a natural adsorbent, due to its high removal efficiencies of the metal ions involved in this study.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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