Kinetic, Isothermal and Equilibrium Studies of the Biosorption of Pb(II) from Solution by a Moss (*Stereophyllum radiculosum*).

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ABSTRACT

In this work, biosorption of Pb(II) ions on *Stereophyllum radiculosum,* a moss, was investigated in batch mode. The equilibrium adsorptive quantity was determined to be a function of the solution pH, contact time, initial concentration of lead ions, and temperature. The study on pH showed that the biosorption is pH dependent. The optimum pH for the biosorption was pH 6. Maximum biosorption was obtained within the first 50 min. of the process. The kinetic study showed that the pseudo-first-order model correlated with the experimental data better than the pseudo-second-order model examined. The biosorption isotherm obtained fitted well into the Freundlich and Langmuir isotherms. The free energy change obtained for the biosorption of Pb(II) at 21^{o}C, initial Pb(II) concentration of 100mgL^{-1} and pH 6 is –9.09 kJmol^{-1}. The high negative value of the standard free energy change (ΔG°) indicates spontaneous nature of the process. The calculated values of ΔH° and ΔS° are negative, showing that the process is exothermic and the degree of disorder reduces with increase in temperature, respectively.

(Keywords: biosorption, Pb(II), *Stereophyllum radiculosum,* isotherm, kinetics, thermodynamics)

INTRODUCTION

The increase in the contamination of the aquatic system with heavy metals through industrial activities has initiated the search for economic strategies for their removal. The pollution of water due to heavy metals is an issue of great environmental concern (Vasuderan et al., 2003).

The heavy metal ions are detected in the waste streams from mining operations, tanneries, electronics, electroplating, batteries and petrochemicals industries. They have harmful effects on human physiology and other biological systems when they exceed the tolerance levels (Kobya, 2004). Exposure to lead can cause anaemia, diseases of the liver and kidney, brain damage, and ultimately death (Jain et al., 1989). It is therefore necessary to remove these heavy metals from industrial effluents.

Major lead pollution can occur through automobiles and battery manufacturing. The conventional methods such as ion exchange, chemical precipitation, redox, and osmosis have been found to be expensive. Agricultural materials have been found to adsorb metal ions hence their application in the removal of heavy metals from solutions (Akar and Tunali, 2006; Liu et al., 2006; Solisio et al., 2006; Babarinde et al., 2007; Chergui et al., 2007; King et al., 2007; Babarinde et al., 2009). In particular, biosorption of lead has been investigated with agricultural materials that have proved effective (Anayurt et al., 2009; Babarinde et al., 2010a, Babarinde et al., 2010b Munagapati et al., 2010; Ngah and Fatinathan, 2010).

The materials usually are composed of lignin and cellulose as major constituents and may also include other polar functional groups of lignin, which includes alcohol, aldehydes, ketones, carboxylic, phenolic and ether groups (Demirbas, 2008). These groups have the ability to some extent to bind heavy metals by donation of an electron pair from these groups to form complexes with the metal ions in solution. The major merits of biosorption technology are the effectiveness in reducing the concentrations of heavy metal ions to low levels and the inexpensive nature of the materials because they are readily available at little or no cost.

Recent studies have shown that *S. radiculosum* is a moss that has great potential for the biosorption of Cd(II) (Babarinde et al, 2008). The aim of the present study was to investigate the possible use of *S. radiculosum* as an alternative biosorbent material for the removal of Pb(II) from solution. The study was therefore aimed at determination of the suitability of *S. radiculosum* in the treatment of industrial effluents containing Pb(II) since other biomass have proved effective in the biosorption of Pb(II) from solution (Babalola et al., 2008; Babarinde et al., 2009, 2010a, 2010b).

The study includes an evaluation of the effects of various parameters such as pH, contact time, initial metal ion concentration, and temperature. The kinetic models and equilibrium isotherm models as well as the thermodynamic parameters related with the process were performed.

MATERIALS AND METHODS

Materials

The *S. radiculosum* is a pleurocarpous (creeping) moss commonly found growing on tree bark near the stem bases of trees in tropical rain forest in different parts of the world. The sample used in this study was collected from a palm tree trunk in a palm tree plantation at Apoje farm settlement in Ogun State, Nigeria. It was rinsed several times with water to remove sand, dried in the oven at 60^oC and ground to powder with mortar. The particles obtained were sieved with 100 mesh size. The biomass was kept in a dry place till the time of usage. Aqueous Pb(II) solution was prepared from analytical grade of $Pb(NO_3)_2$.

The stock solution was prepared with deionized water at the initial concentration of $1000mgL^{-1}$ of Pb(II). The solutions used for the study were obtained by dilution of the stock solution to the required concentrations. The initial pH of each solution was adjusted to the optimum pH value by the drop wise addition of 0.1M HNO_3 and /or 0.1M NaOH solution, except for the study on the effect of pH where the study was carried out at different pH values. Fresh dilution of the stock solution was done for each biosorption study.

Biosorption Studies

Each batch biosorption study was carried out by contacting the biomass with the metal ions under different conditions for a period of time in a glass tube. The uptake capacity of Pb(II) in batch system was studied in this work. Studies were conducted at 27^oC to determine the effect of initial solution pH, contact time, and initial metal ion concentration on the biosorption process. The biosorption process was also monitored within the temperature range $21\text{-}27^oC$ to determine the effect of temperature on the biosorption of Pb(II). Each study was conducted in a thermostated water bath (Haake Wia Model). The water current generated by the pumping in the bath provided the needed shaking of the mixture. The residual metal ions in the supernatant were analyzed using Atomic Absorption Spectrophotometer (Buck Scientific Model Alpha 4 serial number 4200).

Effect of pH on Biosorption

The effect of pH on the biosorption of Pb(II) was carried out within the range that would not be influenced by hydrolysis and precipitation of the lead ions (Pavasant et al., 2006). The suitable pH range for the sorption of Pb(II) has been found to be 1-7.5. Studies were conducted at 27^oC to determine the effect of initial solution pH on the biosorption of Pb(II) by contacting 0.2g of the *S. radiculosum* with 25ml of $100mgL^{-1}$ Pb(II) solution in a glass tube. The pH of each solution had been adjusted to the desired value with 0.1M HNO_3 and /or 0.1M NaOH. The studies were conducted at pH values of 1-7. The glass tubes containing the mixture were left in a water bath for 24 hours. The biomass was removed from the solution by decantation and the residual Pb(II) concentration in the supernatant was analysed. All studies were repeated and the mean value was determined for each pH.

Effect of Contact Time on Biosorption

Initially, studies were conducted at pH 6 to determine the time needed for the biosorption process to reach equilibrium. The biosorption of Pb(II) by *S. radiculosum* was studied at various time intervals (0 – 300 min) at the concentration of $100mgL^{-1}$. 0.2g of the biomass was weighed into each glass tube and 25ml of Pb(II) solution at pH 6 introduced into it. The mixture was left for the different time intervals. The biomass was removed by decantation at the end of each time and the amount of ions in the supernatant was then analyzed.

Effect of Initial Pb(II) Concentration on Biosorption

Batch biosorption study was carried out using a concentration range of 10 – 100mgL^{-1} by introducing 0.2g of the *S. radiculosum* into each of the glass tubes employed and 25ml of Pb(II) solution at pH 6 was added to each of the tubes. The biosorption mixture was then left in a thermostated water bath to maintain the temperature of 27^{o}C for optimum contact time required to reach equilibrium. The *S. radiculosum* was removed from the solution by decantation and the concentration of residual Pb(II) ions in each solution was determined. The amount of metal ions biosorbed from solution was calculated based on the difference on the metal ion concentration in aqueous solution before and after the biosorption process. The removal efficiency (E) of the Pb(II) by the biomass was calculated as:

$$E = 100\left(\frac{C_i - C_e}{C_i}\right) \qquad \textbf{(1)}$$

Where C_i is the initial metal ion concentration (mgL^{-1}) and C_e is the equilibrium metal ion concentration (mgL^{-1}). Analysis of the relationship between biosorption capacity of the biomass and metal ion concentration was performed using the Freundlich (Freundlich ,1906) and Langmuir (Langmuir,1918) isotherms. The linearized form of the Freundlich isotherm is:

$$\log\Gamma = \frac{1}{n}\log C_e + \log K \qquad \textbf{(2)}$$

Where $\frac{1}{n}$ and K are Freundlich characteristic constants, biosorption intensity and biosorption capacity, respectively.

The Langmuir isotherm model assumes monolayer biosorption on the surface with a finite number of identical sites, that all sites are energetically equivalent and that there is no interaction between biosorbed molecules. The linearized form of the Langmuir isotherm is:

$$\frac{1}{\Gamma} = \frac{1}{b_m}\frac{1}{C_e} + \frac{1}{\Gamma_m} \qquad \textbf{(3)}$$

Where b_m is a coefficient related to the affinity between the sorbent and sorbate and Γ_m is the maximum sorbate uptake under the given condition.

Effect of Temperature on Biosorption

The biosorption of Pb(II) by *S. radiculosum* was studied at various temperatures between 21^{o}C and 27 ^{o}C at a constant concentration of 100 mgL^{-1}. 0.2g of the moss was weighed into the tubes and 25ml of solution at the optimum pH 6 was introduced into each tube. The biosorption mixture was then left in a water bath to maintain the temperatures for optimum contact time required to reach equilibrium. The moss was removed from the mixture by decantation at the end of each process.

Statistical Analysis

The curve fittings of all the data obtained were performed using Microcal Origin 6.0 software.

RESULTS AND DISCUSSION

Effect of Solution pH on Biosorption

The result of the pH study is shown in Figure 1. The result shows that maximum biosorption was obtained at pH 6. The percentage Pb(II) biosorption is lower at other pH values. Subsequent biosorption studies were conducted at pH 6. This result confirms the earlier report that biosorption of heavy metals by biomass is pH dependent (Han et al., 2006). The result suggests that optimum biosorption is obtained at pH 6 and that initial pH would play a vital role in the removal of the Pb(II) from aqueous solution using S. *radiculosum*.

At pH values above the isoelectric point, there is a net negative charge on the cell wall components and the ionic state of the ligands will be to such as to promote reaction with metal cations. The major binding groups for biosorption are hydroxyl, carbonyl, carboxyl, sulphydryl, sulfonate, thioether, amine, secondary amine, amide, imine, imidazole, phosphonate, and phosphodiether (Volesky, 2007).

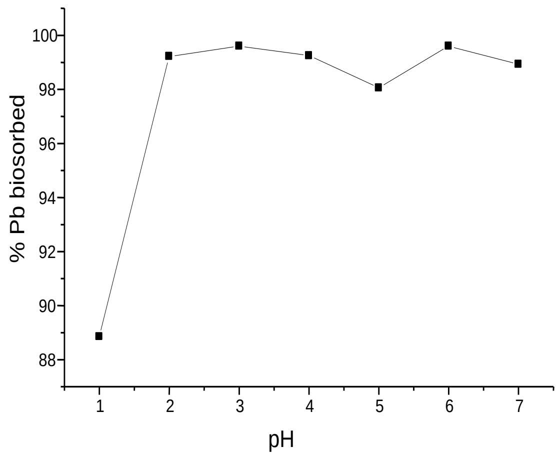

Figure 1: Effect of pH on the Biosorption of Pb(II) by *Stereophyllum radiculosum* at 27°C.

On lowering the pH of the solution, the net charge on the surface of the cell wall will be positive, consequently, there is repulsion on the approach of the positively charged lead ions. This results in the reduction in the amount of lead ions biosorbed by the moss. The pH control in the system is important because it affects both the configuration of the active ion-exchange sites as well as the ionic state of the sorbate in solution (Volesky, 2007). Moreover, depending on the pH values of the aqueous solution, the functional groups participate in metal ion binding (Munagapati et al., 2010). At low pH, the concentration of protons is high and the ion-exchange sites on the biomass become solidly protonated. This, on the other hand, indicates the possibility of displacing the metals sequestered on the biosorbent by a simple acidic wash. The regeneration of the biosorbent material enables its multiple re-use, hence, further increasing the economy of its use.

Biosorption Kinetics

As shown in Figure 2, the biosorption of Pb(II) onto the moss was rapid for the first 30 min and equilibrium was reached within 50 min. The period of 50 min was therefore considered as the optimum time.

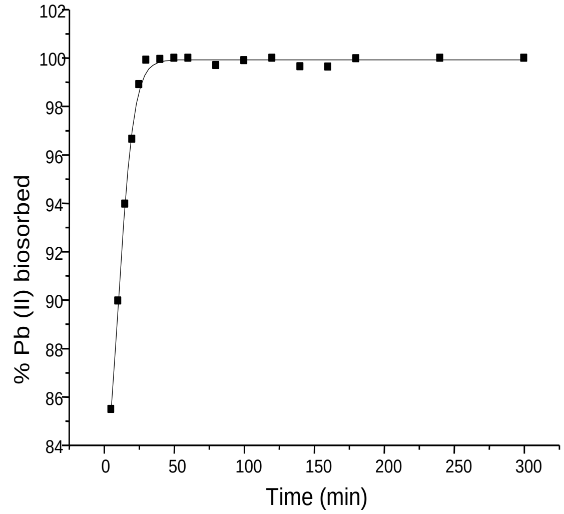

Figure 2: Time Courses of Pb(II) Biosorption by *Stereophyllum radiculosum* 100 mg L^{-1} at 27°C and pH 6.

The reaction involved the biosorption of metal ion (represented as M^{2+} for a divalent metal ion) from the liquid phase to the solid phase, the adsorbent with lone pair of electron (represented as Ä), and can be considered as a reversible reaction with an equilibrium being made between the two phases as schematically shown below:

$$\ddot{A} + M^{2+} \rightleftharpoons A\text{-}M \qquad \textbf{(4)}$$

The pseudo-first-order and pseudo-second-order rate equations (Goel et al., 2005; Kavitha and Namasivayam, 2007) were applied to the kinetic study as shown below in equations (5) and (6), respectively.

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303}t \qquad \textbf{(5)}$$

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

where k_1 is the Lagergren rate constant of the biosorption (min^{-1}); k_2 is the pseudo-second - order rate constant (g mg^{-1} min^{-1}); q_e and q_t are the amounts of metal ions sorbed (mg g^{-1}) at equilibrium and at time t, respectively. The plot of $log(q_e-q_t)$ versus t for the biosorption of Pb(II) ions on the biomass at initial concentration of 100 mg L^{-1} fits the pseudo-first-order reversible kinetics model. The overall rate constant of the biosorption (k_1) was calculated from the slope of the plot.

As shown in Figure 3, the pseudo-first-order equation describes the biosorption process because negative value of rate constant was obtained for the pseudo-second-order equation. Therefore, the biosorption kinetics could well be approximated more favourably by pseudo-first-order kinetic model for Pb(II). Lagergren constants obtained for this study are presented in Table 1. The high value of correlation coefficient obtained shows that the data conforms well to the pseudo-first-order kinetic model. The q_e and k_1 obtained in this study are comparable to those earlier reported (Goel et al., 2005).

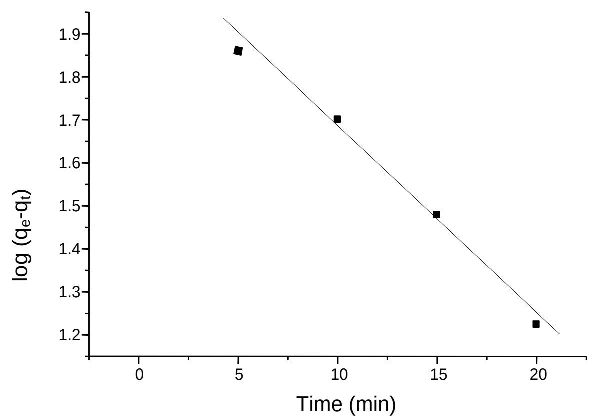

Figure 3: Pseudo-First-Order Kinetic Plot for the Biosorption of Pb(II) by *Stereophyllum radiculosum* at 27°C and pH 6.

Effect of Initial Pb(II) Concentration on Biosorption

The effect of initial Pb(II) concentration on the biosorption capacity shows that up to 82% of the metal ion was biosorbed at the initial metal ion concentration of 100mgL^{-1} within the first 30 min. In Figure 4, the efficiency increases as the initial metal ion concentration increases. The gradual increase in the efficiency of the biomass at later times shows approach to saturation of the available binding sites on it. Such behavior can be attributed to the maintenance of fixed number of binding sites at a given dosage while increasing the concentration of the metal ion.

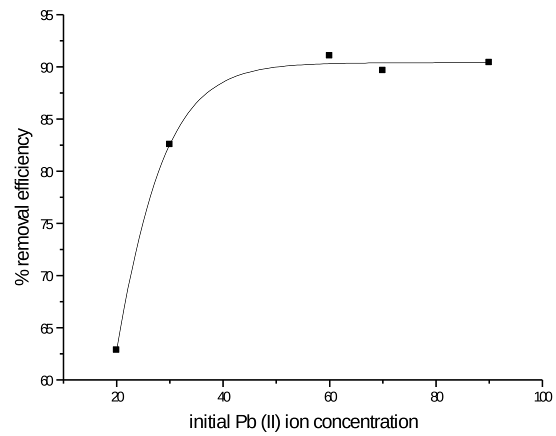

Figure 4: Efficiency Plot for Biosorption of Pb(II) by *Stereophyllum radiculosum* at 27°C and pH 6.0.

Biosorption Isotherm Models

The empirical Freundlich isotherm does not indicate a finite uptake of the biosorbent. The relationship can be reasonably applied to the low or intermediate concentration ranges (Venkateswarlu et al., 2007). The same experimental data were applied to both the Freundlich and Langmuir isotherm models as shown in Figures 5 and 6, respectively.

The Freundlich equation obtained is:

$\log \Gamma = 0.7472 \log C_e + 0.8632$,

while Langmuir equation obtained is:

$1/\Gamma = 5.7205/Ce + 158.73$.

The correlation factors are 0.9988 and 0.9971, for Freundlich and Langmuir isotherms, respectively. The values of the correlation factors indicate good fit. Isothermal biosorption parameters for these isotherms are shown in Table 2. These Freundlich and Langmuir parameters compare well with those of other biosorbents that have been reported (Venkateswarlu et al., 2007). The values of the parameters show that *S. radiculosum* is a good biosorbent for the uptake of Pb(II) from solution.

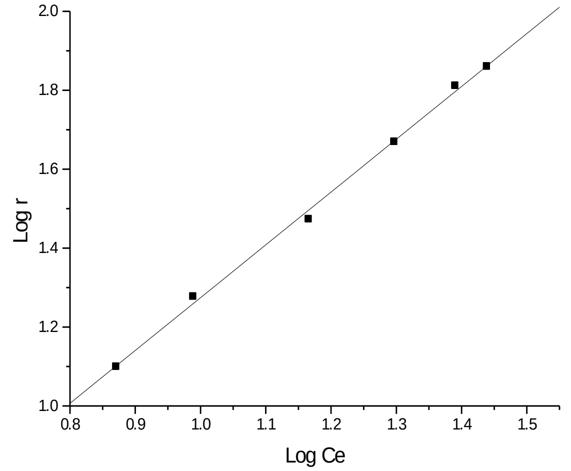

Figure 5: Freundlich Isotherm for the Biosorption of Pb(II) ions by *Stereophyllum radiculosum* at 27^{o}C and pH 6.0.

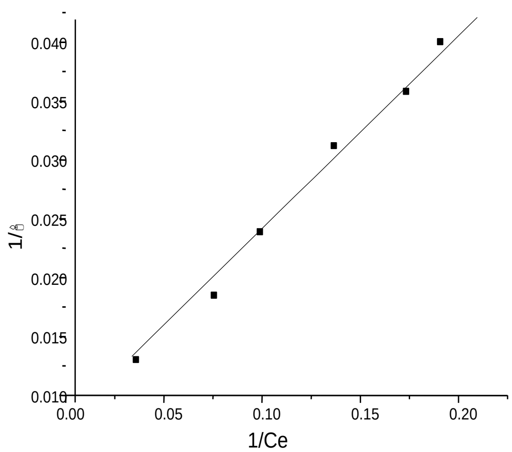

Figure 6: Langmuir isotherm for the biosorption of Pb(II) ions by *Stereophyllum radiculosum* at 27^{o}C and pH 6.0.

Thermodynamics of Pb(II) Biosorption

The experimental data obtained at different temperatures were used in calculating the thermodynamic parameters (ΔH^o, ΔG^o, and ΔS^o) of sorption utilizing the following well-known equations:

$$\Delta G^o = - RT\ln K_c \qquad \textbf{(7)}$$

Where ΔG^o is the standard Gibbs free energy change for the biosorption ($Jmol^{-1}$), R is the universal gas constant (8.314 $Jmol^{-1}k^{-1}$) and T the temperature (K). The distribution ratio, K_c (mLg^{-1}), is defined as:

$$K_c = \frac{C_{ad}}{C_e} \qquad \textbf{(8)}$$

It reflects the extent of distribution of the adsorbate ions between the solid and liquid phases at equilibrium since the biosorption process has been reported as a heterogeneous and reversible process at equilibrium (Han et al, 2005). The change in free energy of the process is related to the changes in enthalpy and entropy of the biosorption process by:

$$\Delta G^o = \Delta H^\circ - T\Delta S^\circ \qquad \textbf{(9)}$$

The free energy change obtained for the biosorption of Pb(II) at 21^{o}C, initial Pb(II) concentration of 100mgL^{-1} and pH 6 is –9.09 $kJmol^{-1}$.

Table 1: Pseudo-First Order Kinetic Model Parameters for the Biosorption of Pb(II) *by Stereophyllum radiculosum* at 27^{o}C and pH 6.

q_e (mg g^{-1})	k_1 (L min^{-1})	R^2	S. D.
499.95	0.098	0.9901	0.0336

Table 2: Freundlich and Langmuir Isothermal Biosorption Parameters for the Biosorption of Pb(II) by *Stereophyllum radiculosum* at 27^{o}C and pH 6.0.

Freundlich Parameters				Langmuir parameters			
$\frac{1}{n}$	K	R	S.D	b_m	Γ_m	R	S.D
1.3383	0.8632	0.9988	0.016	5.7205	158.73	0.9971	0.001

The high negative value of the calculated standard free energy change (ΔG^o) depicts that the biosorption process is largely spontaneous, driven towards the right-hand side. This result validates the feasibility of the biosorption process, and the spontaneity of the biosorption of Pb(II) by *S. radiculosum.*

The effect of temperature on change in free energy obtained for the biosorption of Pb(II) is shown in Figure 7. The 'apparent' values of ΔG^o, ΔH^o and ΔS^o were obtained from the linear plot in Figure 7. The changes in enthalpy (ΔH^o) and entropy (ΔS^o) for the process were obtained by plotting ΔG^o against T as shown in Figure 7. Analysis of the plot shows that ΔH^o is –335.84 kJ mol^{-1} while the ΔS^o is –1.11 kJ $mol^{-1}K^{-1}$. The negative value obtained for ΔH^o demonstrates that the sorption process is exothermic in nature. This behaviour indicates that lower temperature is preferred for higher biosorption of the Pb(II). Similar inverse relationship between the temperature and the spontaneity of the biosorption of Pb(II) has been reported with macrofungus (*Lactarius scrobiculatus*) (Anayurt et al., 2009) and *Acacia leucocephala* bark powder (Munagapati et al., 2010)

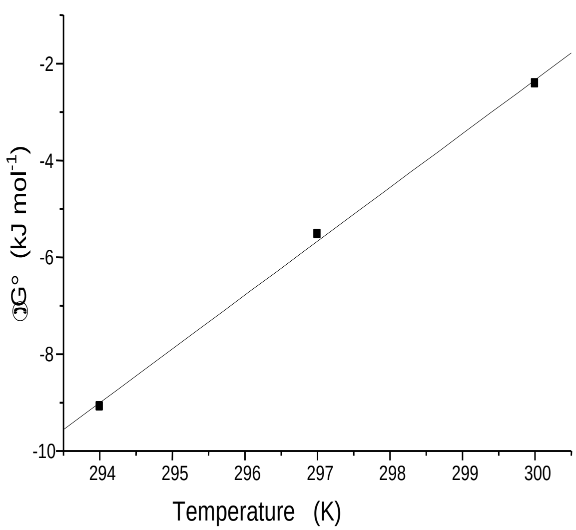

Figure 7: The Free Energy Change for the Biosorption of Pb(II) by *Stereophyllum radiculosum* at 27^{o}C and pH 6.0.

The calculated value has been described as 'apparent' value because it includes plausible energy contribution from the adsorbent and forces of dehydration of the adsorbate ions in addition to the energy associated with the intrinsic adsorption step (Şeker et al., 2008). The calculated value of ΔS^o refers to the entropy change of the biosorption process and not the total entropy change, which can be positive or negative in value. As shown above, negative value of ΔS^o obtained in this study implies less disorder was associated with the biosorption process. Positive value of ΔS^o has been reported to be associated with increase in the dehydration steps of the adsorbate ions, which are known to possess relatively high energies of solvation thereby resulting in the stabilization of water sheaths in the absence of the adsorbent. The implication of the negative value of ΔS^o obtained in this study is that there would be a decrease in the number of steps involved in the biosorption of Pb(II) by *Stereophyllum radiculosum.*

CONCLUSIONS

This study shows that *Stereophyllum radiculosum* could be used as a biosorbent in the treatment of industrial waste water containing Pb(II). The batch studies have shown that the biosorption of Pb(II) is pH dependent and the optimum pH for the removal is 6. The amount of Pb(II) biosorbed increased with increase in initial metal ion concentration. The initial Pb(II) concentration and temperature also influenced the adsorptive quantity. The process of biosorption has nearly reached equilibrium in 30 min. The Freundlich and Langmuir isothermal parameters obtained compare well with those already reported. These results indicate that the *Stereophyllum radiculosum* has potential for the uptake of Pb(II) from industrial effluents.

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