

Equilibrium Modeling and pH-Dependence of Biosorption of Ni(II) and Cr(III) from Solution by Siam Leaf (*Chromolaena odorata*).

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ABSTRACT

The biosorption of Ni(II) and Cr(III) from dilute aqueous solution using Siam (*Chromolaena odorata*) leaf as the biosorbent is reported in this paper. The FT-IR analysis of Siam leaf biomass revealed the presence of functional groups with lone pair of electrons in the cell wall of the biomass. The effects of pH, contact time, initial metal ion concentration, and temperature were determined and each was found to affect the process. Batch biosorption kinetics, equilibrium and thermodynamic experiments were conducted to determine the adsorption kinetic rate constants, the isotherms, and thermodynamic parameters for the biosorption of each of the metal ions. Kinetic studies show that the biosorption of Ni(II) and Cr(III) can be described by pseudo-second-order and pseudo-first-order kinetic models, respectively. Freundlich and Langmuir adsorption models were equally tested. Thermodynamic results indicate that the biosorption of each metal ion by Siam leaf biomass is spontaneous and endothermic.

(Keywords: biosorption, Ni(II), Cr(III), nickel, chromium, adsorption isotherms, Siam leaf)

INTRODUCTION

The fact that heavy metal ions are highly toxic, non-biodegradable, and tend to bio-accumulate causing different health problems in living organisms has made their removal from the environment a global issue. Ni(II) and Cr(III), among other toxic metals, are discharged into the environment from industries such as mineral processing, paint formulation, tanning and electroplating. The undesirable effects of these toxic metals can be controlled by treatment of the industrial effluents containing them before they are discharged into the aquatic environment.

The conventional methods of heavy metal removal from the environment are either economically expensive or technically problematic to handle. These limitations have led to the development of biosorption which removes heavy metal effectively at little or no cost. The process involves the binding and concentration of heavy metals from aqueous solutions by certain types of biomass of plant or animal origin which have functional groups to bind them.

Several materials have been used as biosorbents in the removal of toxic metal ions from solutions (de la Rosa et al, 2008; Anayurt et al, 2009; Babalola et al, 2009; Babarinde et al, 2009a, 2009b; Bankar et al, 2009; Fereidouni, 2009; Gok and Aytas, 2009; Gundogdu et al, 2009; Lakshmanraj et al, 2009; Liang et al, 2009; Mohan and Gandhimathi, 2009; Nadeem et al, 2009; Naiya et al, 2009a, 2009b; Qaiser et al, 2009; Vimala and Das, 2009; Okasha and Ibrahim, 2010; Senthilkumar, et al, 2010; Tuzen and Sari, 2010; Vijayaraghavan, et al, 2010). The results of the different studies have shown that this process is both effective and efficient in treating solutions containing toxic metal ions. The objective of this study was to investigate the biosorption of Ni(II) and Cr(III) by Siam leaf biomass from aqueous media.

Siam leaf (*Chromolaena odorata*) is a species of flowering shrub in the sunflower family, Asteraceae. It is native to North America, from Florida and Texas to Mexico and the Caribbean, and has been introduced to tropical Asia, west Africa, and parts of Australia. Common names include Siam Weed, Christmas Bush, and Common Floss Flower. It is sometimes grown as a medicinal and ornamental plant. It is used as a traditional medicine in Indonesia.

MATERIALS AND METHOD

Biomass Preparation

The leaves used for this study were obtained from sites within the campus of Olabisi Onabanjo University, Ago-Iwoye, Ogun State, Nigeria. The leaves were rinsed with deionized water to remove debris, sun dried and cut into pieces of approximately 0.5cm. The samples were kept dry until the time of usage.

Reagents and Equipment

All chemical reagents used in this study were of analytical grade. Deionized water was used for preparation and dilution of all solutions. Ni(II) and Cr(III) stock solutions were prepared as 1000 mgL⁻¹ by dissolving appropriate amount of Ni(NO₃)₂·6H₂O and Cr(NO₃)₂·9H₂O in a 1-liter standard flask using deionized water. The stock solutions were diluted to the required concentrations and appropriate pH with a dropwise addition of 0.1 M HNO₃ and /or 0.1 M NaOH using a pH meter. Freshly diluted stock solutions were used for each experiment. Atomic Absorption Spectrophotometer (AAS) (Buck scientific model 210 VGP) was used for analysis of residual metal ions in solution after each biosorption process. Fourier Transform Infrared (FT-IR) spectra of unloaded and metal-bound Siam leaf samples were recorded using an FT-IR spectrophotometer (Shimadzu FT-IR 8400S).

Batch Biosorption Procedure

Biosorption was carried out using batch method. In order to optimize the experimental conditions, the batch biosorption studies were performed for different pH, contact times (0 - 300 min), metal concentrations (10 - 100 mgL⁻¹), and temperature (27-50°C). Each batch biosorption study was carried out by contacting 2.5 g of the Siam leaf with 25 ml of a given concentration of metal ion solution in a thermostated water bath (Haake Wia model) for temperature regulation. In each case, a parameter was varied whilst others were kept constant. At the end of the contact time, the solution was decanted into sample bottle and analyzed for the residual metal ions in solution using AAS. Each experiment was repeated and the mean value calculated.

Statistical Analysis

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

RESULT AND DISCUSSION

FT-IR Analysis

The FT-IR spectroscopy method was used to obtain information about the nature of the functional groups responsible for the biosorption of the metal ions on the binding sites of the Siam leaf (Elangovan et al, 2008; Ertugay and Bayham, 2008; Bueno et al, 2008). Figure 1 is a sample of the FT-IR spectra for the unloaded and metal-loaded Siam leaf.

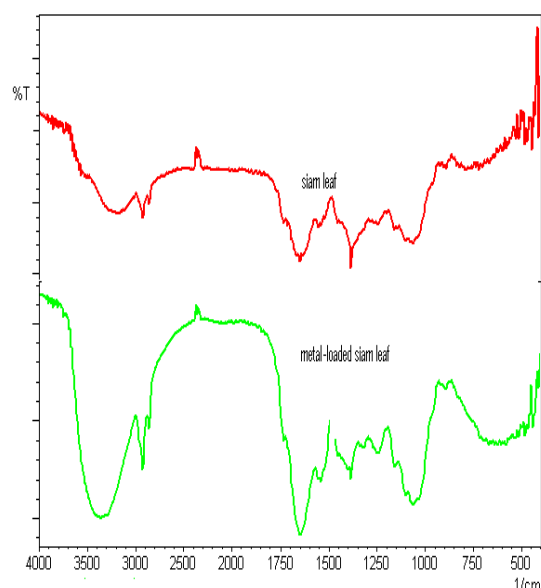


Figure 1: FT-IR Spectra of the Unloaded and Metal-Loaded Siam Leaf Biomass.

The functional groups responsible for the bands are shown in Table 1. The IR spectra indicate that the cell wall of Siam leaf possesses different surface structures and functional groups with lone pairs of electron that are available to bind to the positively divalent or trivalent metal ions. The reaction involved the biosorption of metal ion (represented as M²⁺ for a divalent metal ion) from the liquid phase to the solid phase, the adsorbent with lone pair of electron (represented as Å), and can be considered as a reversible reaction with an equilibrium being made between the two phases as schematically shown in Equation (1).

Table 1: IR Absorption Bands and the Corresponding Possible Functional Groups.

Wave number (cm ⁻¹)	Functional group
3300-3400	OH, NH stretch
2920-2850	C – H stretch
2360.95	O – H carboxylic acids
2121.77	C ≡ C
1710-1690	C = O
1519.96-1516.10	N = O Nitro (R – NO ₂)
1423.51-1431.23	C – H bend
1234.48-1246.06	C – X
1050	S – O sulphuroxides
895.00	C – H Aromatic (out of plane)
783.13-775.41	C – H Aromatic (out of plane)



Influence of pH

The initial pH of the solution has been reported as one of the most important parameters in the biosorption of metal ions by biomass (Anayurt et al, 2009). The pH determines the net charge on the biomass and this invariably determines whether the metal ions can bind or not. As shown in Equation (1), biosorption process is reversible in the presence of mineral acid because the net charge on the biomass can be regulated. This is why desorption of the already biosorbed metal ions is possible (Anayurt et al, 2009). The effect of pH on biosorption of Ni(II) and Cr(III) onto Siam leaf is shown in Figure 2. The results show that maximum biosorption was obtained at pH 3 for Cr(III) and pH 5 for Ni(II) hence, subsequent studies were carried out at those pH values which serve as the optimum pH values.

The mechanism of biosorption on the surface of the biomass is a reflection of the physicochemical interaction of the species in solution and the biosorptive sites of the sorbent (Aksu et al., 2002; Amini et al, 2008). At low pH, the solution is highly acidic, consequently, the net charge on the active sites is positive. Also, the metal ions compete with protons for the active sites on the cell wall of the biomass. Ultimately, the result is reduction in the uptake of the metal ions by the biosorbent. In addition, the protonation of the active sites on the cell wall of the biomass reduces the number of negatively charged sites needed for the metal ions to bind. As the pH increases, the functional groups in the cell wall

become deprotonated and free for the metal ions to bind. Therefore, the competition between metal ions and protons is reduced, thus the increase in the biosorption with increase in pH. Similar results have been reported for different biomass in the biosorption of Cd(II) and Pb(II) (Anayurt et al., 2009; Vimala and Das, 2009; Babarinde and Babalola, 2010).

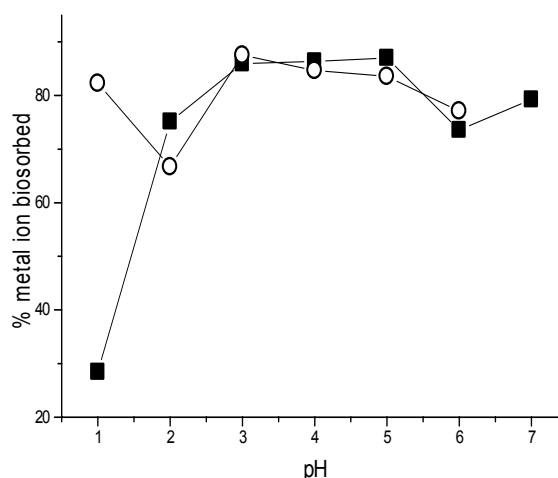


Figure 2: Effect of pH on the Biosorption of Ni(II) (squares) and Cr(III) (circles) by Siam Leaf (*Chromolaena odorata*).

The Influence of Contact Time

The biosorption of Ni(II) and Cr(III) by Siam leaf as a function of time is shown in Figure 3.

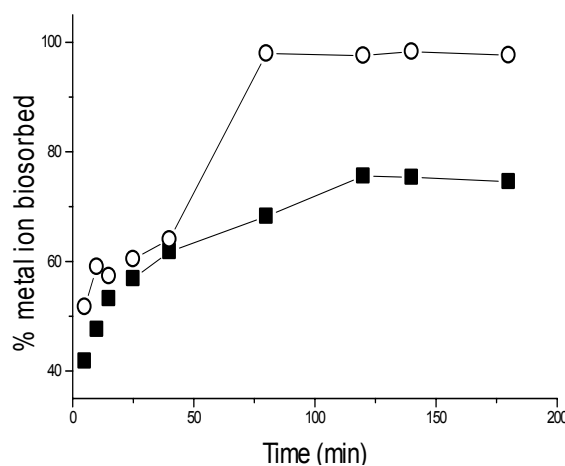


Figure 3: Time Course for the Biosorption of Ni(II) (squares) and Cr(III) (circles) by Siam Leaf (*Chromolaena odorata*) at Temperature of 300K.

The biosorption of the two metal ions from solution increased rapidly with increase in time within the first 30 min. After 80 min the sorption of Cr(III) has reached the maximum level, hence 80 min was taken as the optimum time for the biosorption of Cr(III). The rapidity in the biosorption of the ions from solution at the initial state reflects the availability of abundant active binding sites on the cell wall of the biomass. In the case of Ni(II), maximum sorption was obtained after 120 min. This shows that the sorption of Cr(III) is faster than that of Ni(II). This can be accounted for by the higher valence of the former. The rate of biosorption is in the order Ni(II) < Cr(III) which is the order of increasing hydrated ionic size (Kielland, 1937). The difference can be accounted for in terms of the differences in the order of hydrated ionic sizes of the metal ions.

The rate of biosorption does not increase after the optimum time due to the fact that the available binding sites on the cell wall were used up therefore, further increase in contact time does not lead to any increase in biosorption of the metal ions.

The results of this kinetic study were tested with three kinetic models. The pseudo-first-order, pseudo-second-order and the intraparticle diffusion equations were applied to the kinetic study as shown in Equations (2) – (4), respectively.

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (2)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (3)$$

$$R = K_s t^b \quad (4)$$

where k_1 is the Lagergren rate constant of the biosorption (min^{-1}); k_2 is the pseudo-second-order rate constant ($\text{g mg}^{-1} \text{min}^{-1}$); q_e and q_t are the amounts of metal ions sorbed (mg g^{-1}) at equilibrium and at time t , respectively. R is the percent metal biosorbed, K_s is the intraparticle diffusion constant, t is the contact time, while b is the gradient of the linear plot as shown in Equation (5).

In the linear form, Equation (4) turns to Equation (5):

$$\log R = b \log t + \log K_s \quad (5)$$

Only pseudo-first-order model fits the biosorption of Cr(III) whilst only the pseudo-second-order model fits the biosorption of Ni(II) by Siam leaf. For the former, a plot of t against $\log(q_e - q_t)$ gives a straight line graph whose slope was used to evaluate the value of and k_1 . The plot of (t/q_t) versus t gives a linear graph for pseudo-second-order kinetics. The pseudo-second-order rate constant (k_2) of the biosorption was calculated from the intercept of the plot. The linearity of the plots of Figures 4a and 4b as indicated by $R^2 \approx 1$ shows the applicability of the models for this biosorption process. Therefore, the kinetic parameters are shown in Tables 2 and 3, respectively.

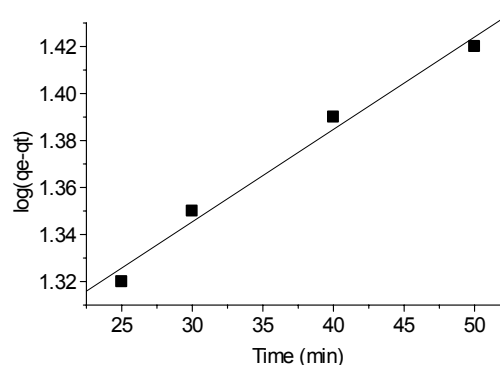


Figure 4a: Pseudo-First-Order Plot for the Biosorption of Cr(III) at pH 3 by Siam Leaf (*Chromolaena odorata*) at Temperature of 300K, pH 3, 100 mg L⁻¹.

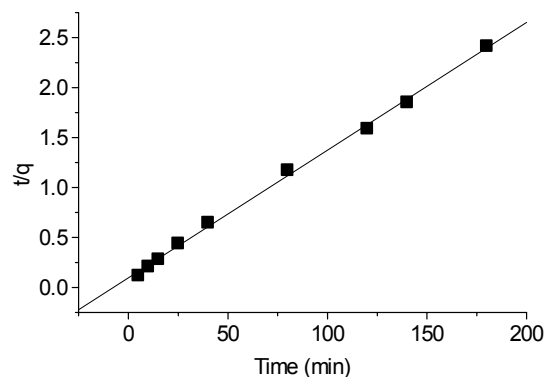


Figure 4b: Pseudo-Second-Order Plot for the Biosorption Ni(II) by Siam Leaf at pH 5, 100 mg L⁻¹, 300K.

Table 2: Pseudo-First-Order Kinetic Model Parameters for the Biosorption of Cr(III) by Siam Leaf at pH 3, 100 mg L⁻¹, 300K.

q _e (mg g ⁻¹)	k ₁ (min ⁻¹)	R	S.D.
16.885	9.0508x10 ⁻³	0.9915	0.007

Table 3: Pseudo-Second-Order Kinetic Model Parameters for the Biosorption of Ni(II) by Siam Leaf at pH 5, 100 mg L⁻¹, 300K.

q _e (mg g ⁻¹)	k ₂ (g mg ⁻¹ min ⁻¹)	R	S.D.
78.31	1.679x10 ⁻³	0.9991	0.03815

The Influence of Initial Metal Ion Concentration

The initial concentration of metal ion in solution determines the amount of the metal ions biosorbed by the sorbent when there are available binding sites. This is evident in the results shown in Figures 5a and 5b.

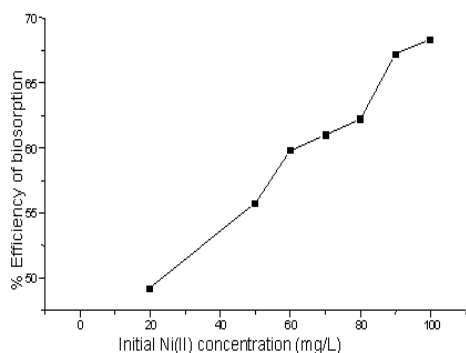


Figure 5a: Efficiency Plot for the Biosorption of Ni(II) by Siam Leaf at pH 5, 100 mg L⁻¹, 300K.

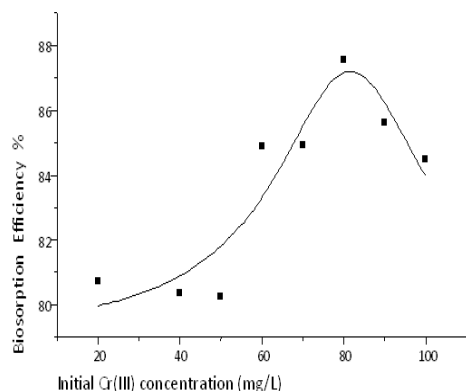


Figure 5b: Efficiency Plot for the Biosorption of Cr(III) by Siam Leaf at pH 3, 100 mg L⁻¹, 300K.

The increase in uptake capacity with increase in initial metal concentration has been accounted for in terms of higher availability of metal ions for sorption (Vimala and Das, 2009). The increase in concentration leads to increase in collision between the metal ions and the sorbent which is a major factor in kinetics for increase in the rate of chemical reactions. The biosorption efficiency for each metal was calculated using Equation (6).

$$E = 100 \left(\frac{C_i - C_e}{C_i} \right) \quad (6)$$

where C_i and C_f are the initial and final metal ion concentrations, respectively. The result is shown in Figures 5a and 5b. This trend of increase in efficiency with increase in initial metal ion concentration is similar to earlier report (Babarinde et al, 2009a). The trend of initial rapid increase in efficiency is due to the availability of binding sites on the biomass which reduce with increase in concentration of metal ions. Consequently, the sorption approached saturation stage as the fixed number of binding sites were exhausted.

Biosorption Isotherm Models

The equilibrium sorption isotherms provide vital data to understand the mechanism of the biosorption process. Out of the different isotherms used to explain the nature of sorbent/sorbate interface in biosorption, the most common of them are Freundlich and Langmuir isotherms. These isotherms are generally used to establish the relationship between the amount of metal ion sorbed and its equilibrium concentration in solution. The Freundlich isotherm model assumes that the removal of metal ions occurs on a heterogeneous adsorbent surface and can be applied to multilayer biosorption of the metal ions. The linearised Freundlich isotherm is expressed in Equation (7):

$$\log \Gamma = \frac{1}{n} \log C_e + \log K_f \quad (7)$$

where K_f is a constant related to the biosorption capacity (mg g⁻¹) and 1/n is an empirical parameter related to the biosorption intensity. The Freundlich isotherms for the biosorption of Ni(II) and Cr(III) are shown in Figures 6a and 6b.

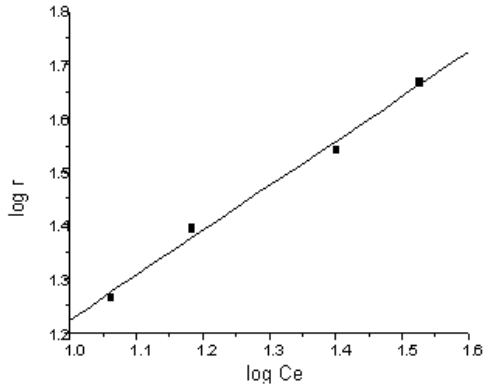


Figure 6a: Freundlich Isotherm for the Biosorption Ni(II) by Siam Leaf at pH5, 300K.

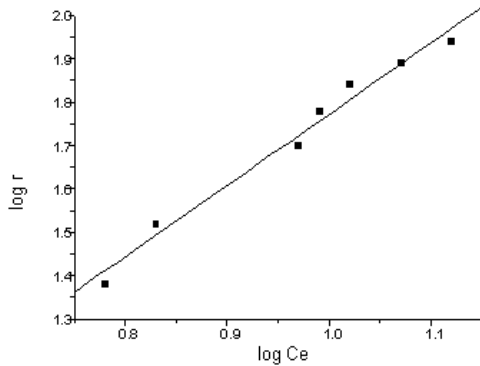


Figure 6b: Freundlich isotherm for the biosorption Cr(III) by Siam leaf at pH3, 300K.

The Freundlich isothermal parameters for this study are given in Table 4. The Langmuir isotherm model is used for monolayer sorption onto a surface containing a finite number of identical binding sites on the sorbent. The linearized form of the Langmuir isotherm model is given in Equation (8).

$$\frac{1}{\Gamma} = \frac{1}{b_m} \frac{1}{C_e} + \frac{1}{\Gamma_m} \quad (8)$$

where b_m is a coefficient related to the affinity between the sorbent and sorbate, Γ_m is the maximum sorbate uptake under the given condition. The Langmuir isotherms for the biosorption of Ni(II) and Cr(III) are shown in Figures 7a and 7b and the Langmuir isothermal parameters are given in Table 4. The fitness of the biosorption data is measured by the value of regression coefficients (R^2) given in Table 4.

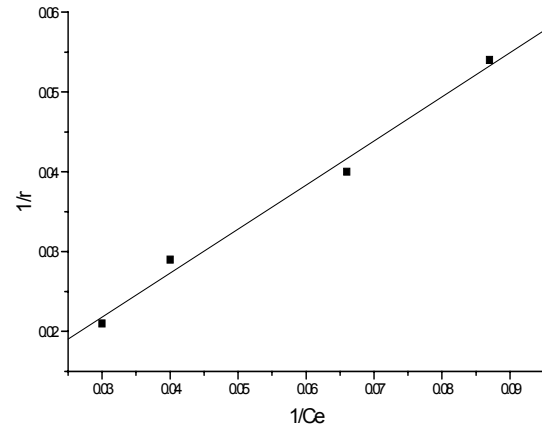


Figure 7a: Langmuir Isotherm for the Biosorption Ni(II) by Siam Leaf at pH5, 300K.

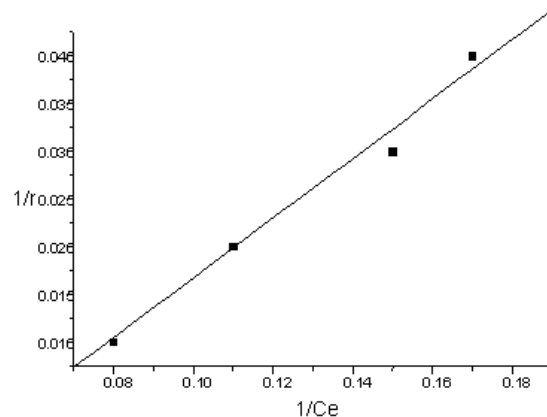


Figure 7b: Langmuir Isotherm for the Biosorption Cr(III) by Siam Leaf at pH3, 300K.

Table 4: Freundlich and Langmuir Isothermal Biosorption Parameters for the Biosorption of Ni(II) [pH 5] and Cr(III) [pH 3] by Siam Leaf at Initial Concentration of 10 - 100 mgL⁻¹, 300K.

Metal ion	Freundlich Isotherm Model				Langmuir Isotherm Model			
	n	K _f	R ²	S.D.	b _m	Γ _m	R ²	S.D.
Ni(II)	1.1980	2.4474	0.9926	0.0184	1.8116	192.31	0.9890	0.0018
Cr(III)	0.6080	1.3440	0.9807	0.0312	3.1452	-64.35	0.9856	0.0019

The Freundlich equations obtained are:
 $\log \Gamma = 0.8347 \log C_e + 0.3887$ and
 $\log \Gamma = 1.6448 \log C_e + 0.1284$, respectively. The Langmuir equations obtained are:

$$\frac{1}{\Gamma} = 0.552 \frac{1}{C_e} + 0.0052 \quad \text{and}$$

$$\frac{1}{\Gamma} = 0.318 \frac{1}{C_e} - 0.0155 \quad \text{respectively.}$$

Thermodynamics of the Biosorption Process

The effect of temperature on the biosorption of the metal ions was investigated and the thermodynamic parameters (changes in standard Gibbs free energy, ΔG° , enthalpy, ΔH° , and entropy change, ΔS°) were calculated using Equations (9) – (11).

$$\Delta G^\circ = -RT \ln K_{ad} \quad (9)$$

$$K_{ad} = \frac{C_{ad}}{C_e} \quad (10)$$

$$\Delta G^\circ = -\Delta S^\circ(T) + \Delta H^\circ \quad (11)$$

where R is the universal gas constant ($8.314 \text{ J mol}^{-1} \text{ K}^{-1}$) and T is the absolute temperature (K). From Equation (11), the values of the changes in entropy, ΔS° , and the enthalpy, ΔH° , are obtained from the slope and the intercept, respectively.

The profiles for the change in free energy with temperature for the biosorption of Ni(II) and Cr(III) are shown in Figures 8a and 8b. In Figure 8a, the ΔH° is $6328.98 \text{ J mol}^{-1}$ while the ΔS° is $21.70 \text{ J mol}^{-1} \text{ K}^{-1}$. In Figure 8b, the ΔH° is $21567.58 \text{ J mol}^{-1}$ while the ΔS° is $78.50 \text{ J mol}^{-1} \text{ K}^{-1}$. The high value of R^2 (≈ 1) obtained for each of the plots shows the agreement between the change in free energy and the temperature of the biosorption process.

The positive values of enthalpy change (ΔH°) for the two sorbates indicate that each of the biosorption process is endothermic. The positive values of change in entropy (ΔS°) implies an increase in the randomness at the solid/solution interface during the biosorption process. The positive values of change in entropy for the biosorption of the metal ions show that the sorption process is highly disordered.

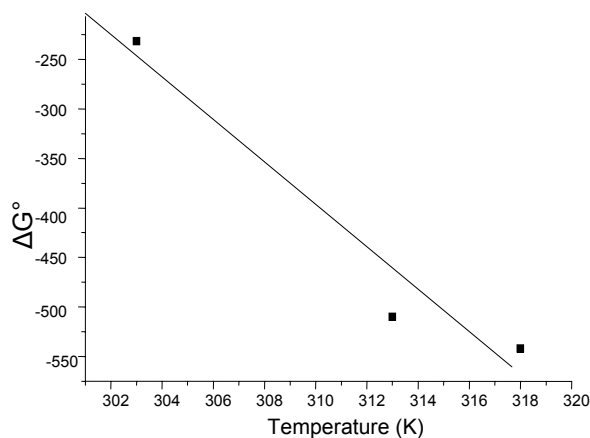


Figure 8a: Change in Free Energy with Temperature for the Biosorption Ni(II) by Siam Leaf at pH 5, concentration of 100 mg L^{-1} , 300K.

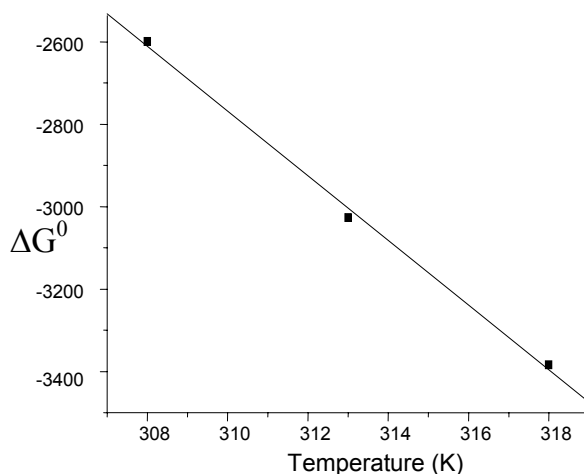


Figure 8b: Change in Free Energy with Temperature for the Biosorption Cr(III) by Siam Leaf at pH 3, Concentration of 100 mg L^{-1} , 300K.

The positive value has been reported to be associated with increase in the dehydration steps of the sorbate ions, which are known to possess relatively high energies of solvation thereby resulting in the stabilization of water sheaths in the absence of the sorbent. The positive values of ΔS° obtained in this study implies that there would be an increase in the number of steps involved in the biosorption of the metal ions by Siam leaf. Similarly, the enthalpy change is positive, indicative of endothermic process. Consequently, it is expected that increase in temperature favors the process.

CONCLUSION

This study shows that Siam leaf has much potential as an efficient and useful biosorbent for the removal of Ni(II) and Cr(III) from wastewaters. The FT-IR study demonstrated the involvement of functional groups with lone pairs of electrons for binding with the metal ions. The pH study shows that the parameter is an important factor in the biosorption of the metal ions from solutions. The kinetic study reveals that pseudo-second-order kinetic model describes the biosorption of Ni(II) whilst the pseudo-first-order kinetic model describes the biosorption of Cr(III). The Freundlich and Langmuir isotherm models fit the biosorption of both metals. The thermodynamic study shows that the process was endothermic for the two metal ions whilst change in entropy of biosorption is positive indicative of increase in disorder at the solid-liquid interface. In each of the cases, the value of ΔG° is negative showing that the process is spontaneous.

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