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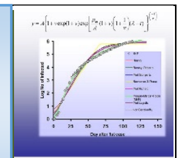

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Mathematical models

Thermodynamic Study on the Biosorptive Removal of Lead (II) Ions from Aqueous Solutions using Acid-treated *Cystoseira tricta* Biomass

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ABSTRACT

Thermodynamic studies on adsorption process play a vital role in the estimation of adsorptive mechanisms (both physical and chemical). Estimating the accuracy of thermodynamic parameters is dependent upon the equilibrium constants between two phases (K_L: dimensionless). In this study, thermodynamic parameters were calculated from the K_L constant derived from the adsorption of Langmuir isotherms of Pb(II) on an sulphuric acid-treated brown algae *Cystoseira stricta* biomass at different temperatures. The conversion of the K_L values to the dimensionless K_c values based on the Langmuir model was then assessed for thermodynamic parameters via the Van't Hoff's equation. The Pb(II) adsorption process onto the *Cystoseira stricta* biomass was spontaneous and feasible with ΔG values at 25, 30, 35 and 40 ^{o}C of -24.56 (95% C.I., -24.00 to -25.03), -22.60 (95% C.I.,-21.95 to -23.12), -20.90 (95% C.I.,-18.87 to -22.01) and -17.17 (95% C.I.,-17.71 to -21.41), respectively, and occurred in an endothermic nature (ΔH= -0.30 kJ/mol (95% C.I., -194.34 to 193.74) with an increased in randomness (ΔS= -299.47 J/mol×K (95% C.I., -553.60 to -45.35) kJ/mol). To confirm the accuracy and precision of the asymptotatic 95% confidence interval, Monte Carlo simulation may be carried out in the future.

INTRODUCTION

Wastes accumulation in the environment results in their dispersion on soils, superficial waters, atmosphere, and particularly aquifers that constitute reservoirs for drinking water. To find a solution to the dispersion of contaminants in the atmosphere, one might think that, remediation being the use of chemical or biological methods for the removal of the polluting substance due to the accidental or intentional discharges. Heavy metals such as copper, zinc, lead, cadmium, chromium, nickel etc., are considered to be major pollutants of industrial activities origin in our environment whose impact depends solely on their behaviour and response to the physicochemical and biological conditions of the medium [1]. The most significant processes that cause the accumulation of the heavy metals on suspended particles and sediments are the adsorption methods and complexes formation [1]. Lead is a heavy metal that is harmful to the human biosystem and is one of the prevalent global contaminants resulting from an increase in industrialization. Accumulation of relatively small quantities of lead in the human body can cause organs malfunctioning and chronic toxicity [2–9].

Biosorption is currently, the most cost-effective method in remediation of pollutants in water bodies [10–17]. The thermodynamics, isotherms, and kinetics play a vital role in understanding the adsorption of organic and inorganic compounds in an aqueous solution. Thermodynamics studies can successfully demonstrate adsorption mechanisms through a series of the adsorptive equilibrium experiments under various degrees of temperatures and different initial concentrations of adsorbate as well as fixed optimal conditions such as pH of the solution, the adsorbent's particle size, ionic strength, and solid or liquid ratio. Thermodynamic parameters such as Gibb's free energy (ΔG°), enthalpy (ΔH), entropy (ΔS) and activation energy (E_a) can be calculated when the adsorption process reaches equilibrium [18].

However, the essential function of dimensionality of the constant K_C thermodynamic equilibrium is often overlooked. As a consequence, a confused representation typically shows both the signs and magnitude of the thermodynamic parameters ΔG^o, ΔH^o, and ΔS^o. The Langmuir constant in its dimensionless form [18] can be used to compute the thermodynamic parameters and this is the objective of this study.

MATERIALS AND METHODS

Data Acquisition

The graphical data of a published work by Iddou et. al. [19] from **Fig. 7**. of Pb(II) sorption on sulphuric acid-treated brown algae at different temperatures modelled according to the Freundlich isotherms was processed using the software Webplotdigitizer 2.5 [20] which digitizes the scanned **figure** and has been use and acknowledged by many researchers because of its precision and reliability. After processing the data, it was remodeled according to Langmuir isotherm model, regressed using nonlinear regression and converted to dimensionless [18].

Calculation on dimensionless equilibrium constant K_L

The Langmuir equation (**equation 1**) was initially derived from a kinetic study and the subsequently from a thermodynamic study. Accurate estimation of thermodynamic parameters is directly dependent on precise analysis of the equilibrium constant between two phases, K_C.

$$q_e = \frac{q_{mL} K_L C_e}{1 + K_L C_e} \qquad \textbf{(Eqn. 1)}$$

Derived K_c from Langmuir was converted to dimensionless form using **equation 2** and being used in **equation 4** as follows [14]:

$$K_C = 10104 \times 55.5 \times 1000 \times K_L \qquad \textbf{(Eqn. 2)}$$

where the fact 55.5 is the number of moles of pure water per liter and the term of 10104 x 55.5 x1000 x K_L is dimensionless. The atomic weight of Pb(II) is 207.2 gmol^{-1}. The relationship of dimensional K_L with unitless equilibrium constant of K_C is described in **equation 3** where, K_L (L/mol) is the Langmuir constant, C° is the selected standard of adsorbate (C°=1 mol/L); γ (dimensionless) is the activity coefficient of adsorbent in solution [15].

$$K_C \approx \frac{K_L\left(\frac{L}{mol}\right) \times C^{\circ}\left(\frac{mol}{L}\right)}{\gamma} \qquad \textbf{(Eqn. 3)}$$

The laws of thermodynamics were applied in the calculation of adsorption thermodynamics parameters (ΔG°, ΔH° and ΔS°) using the van't Hoff equation. When adsorption reaches equilibrium, the free energy change (ΔG) is nearly zero. **equation 3** becomes **equation 5** that has been commonly used to compute ΔG° (standard Gibbs energy change).

$$\Delta G^{\circ} = -RT \ln K_C \qquad \textbf{(Eqn. 4)}$$

$$\ln K_C = \frac{-\Delta H^{\circ}}{R} \times \frac{1}{T} + \frac{\Delta S^{\circ}}{R} \qquad \textbf{(Eqn. 5)}$$

The universal gas constant, R is 0.00831 kJ/mol×K. The relationship between $\Delta G°$, $\Delta H°$ and $\Delta S°$ of an adsorption process is express as follows:

$$\Delta G^{\circ} = \Delta H^{\circ} - T\Delta S^{\circ} \qquad \textbf{(Eqn. 6)}$$

Fitting of the data

Nonlinear regression analysis was carried out using the curve expert professional software (version 1.6). The mean K_L value from Langmuir unitless were obtained for each temperature (25, 30, 35 and 40 °C).

RESULTS AND DISCUSSION

The absorption thermodynamics data from a published work [2] on the biosorption of Pb(II) by sulphuric acid-treated brown algae were analysed using another thermodynamic alternative calculation approach by Nguyen's method [15]. Gathering the information, the units of adsorption isotherms (the plot of q_e against C_e) must be presented as mol/Kg (q_e) and mol/L (C_e) as shown in **Figs. 1** to **4**.

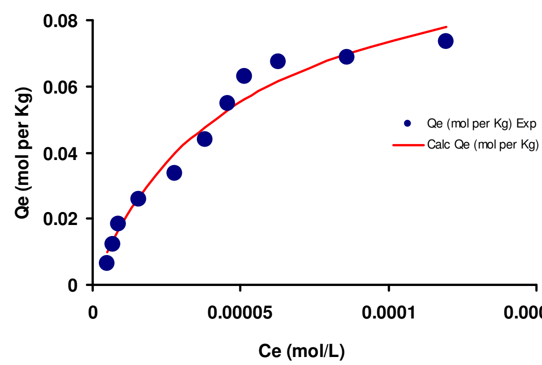

Fig. 1. Equilibrium adsorption isotherm of Pb(II) adsorption onto *Cystoseira stricta* at 25 °C as modelled according to the Langmuir model. Recalculation of the equilibrium constant K_c, into the dimensionless form was utilized from K_L constant of the Langmuir fitting.

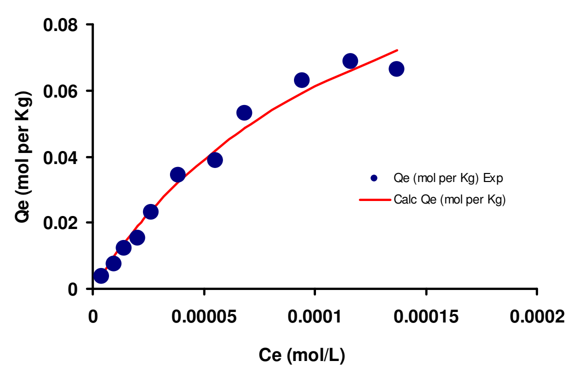

Fig. 2. Equilibrium adsorption isotherm of Pb(II) adsorption onto *Cystoseira stricta* at 30 °C as modelled according to the Langmuir model. Recalculation of the equilibrium constant K_c, into the dimensionless form was utilized from K_L constant of the Langmuir fitting.

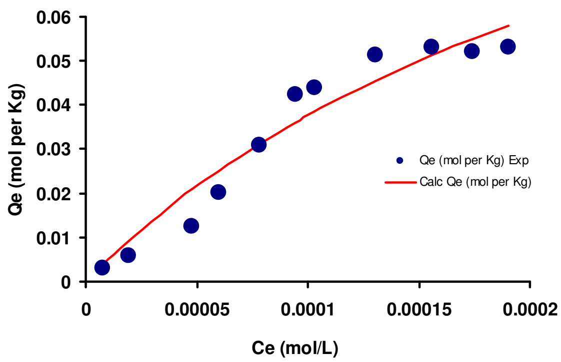

Fig. 3. Equilibrium adsorption isotherm of Pb(II) adsorption onto *Cystoseira stricta* at 35 ºC as modelled according to the Langmuir model. Recalculation of the equilibrium constant K_c, into the dimensionless form was utilized from K_L constant of the Langmuir fitting.

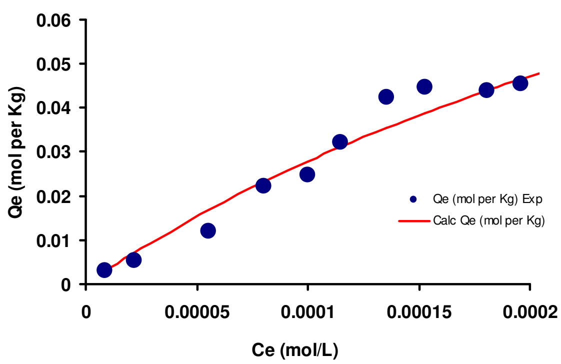

Fig. 4. Equilibrium adsorption isotherm of Pb(II) adsorption onto *Cystoseira stricta* at 40 ºC as modelled according to the Langmuir model. Recalculation of the equilibrium constant K_c, into the dimensionless form was utilized from K_L constant of the Langmuir fitting.

Adsorption Isotherm

In general, adsorption isotherms are used to describe the relationship between an adsorbate at liquid phase (Pb (II) in this case) and adsorbent at solid phase (*Crystoseira stricta* biomass) at a constant temperature under given conditions of pH, ionic strength, fixed mass and particle size of adsorbent [18]. Researches have shown that presenting a plot of Q_e versus C_e of a complete adsorption isotherm in adsorption studies plays a significant role in identifying the region where the experimental equilibrium data are situated [21]. Examples of adsorption isotherms include Henry, Langmuir, Freundlich, BET, etc. in this study, Freundlich isotherm was employed in estimating the thermodynamic parameters.

Studies in isotherms, kinetics, and thermodynamics play an important role in fully understanding the mechanism of adsorption in aqueous solutions of organic and inorganic compounds. Thermodynamic adsorption is important in the three experiments, taking into account the forms and processes of the adsorption process under the temperature variations of the solution. Via a sequence of adsorptive equilibrium tests under various temperatures and different initial adsorbate concentrations and fixed optimum conditions, thermodynamics will effectively exhibit adsorption mechanisms. (pH of the solution, the adsorbent's particle size, ionic strength, and solid/liquid ratio) [18,22–31].

Thermodynamic parameters measure the feasibility and spontaneity of an adsorption method. They are also important for the assessment of adsorbents (i.e. physisorption, ion exchange or chemisorption) [2,7]. The precise calculation of these thermodynamic parameters completely depends on the equilibrium constant of the K_c. In calculating thermodynamic parameters, particularly free energy Gibbs, many authors make use of constants derived from different isothermal models, partition constants and distribution coefficient.

According to the IUPAC, the Gibbs free energy change (after ΔG°) must be determined by a normal balance constant K_c for calculating Gibbs free energy change. The calculated values of K_c (**Table 1**) based on the IUPAC suggestion was carried out according to Tran et al [18] using the Langmuir model as the principal isotherm.

Table 1. Comparison of original Langmuir constant (with unit) and recalculated dimensionless equilibrium constant K_C derived from the Langmuir isotherm replot.

T ºC	K_L(L/mol) (Iddou et al. [19])	K_C (dimensionless). This study (± Std Error)
25	15.3	20220.90±4140.38
30	13.61	7879.26 ±1787.83
35	10.79	3499.55±1913.58
40	7.56	2322.27±1419.19

Table 2 summaries the calculating performance of the parameters of thermodynamics for whole the adsorption process. Based on the van't Hoff plot in **Fig. 5**, the negative values of standard Gibbs free energies (–ΔG°) which do not differ much from the original data suggest that the process of Pb(II) adsorption onto brown algae occurred spontaneously without the requirement of energy or heat. The conversion to the dimensionless form gives ΔG° values that indicated the adsorption occurred through chemisorption as adsorption with ΔG° values ranging from −400 to −80 kJ/mol corresponds to chemisorption [7,12].

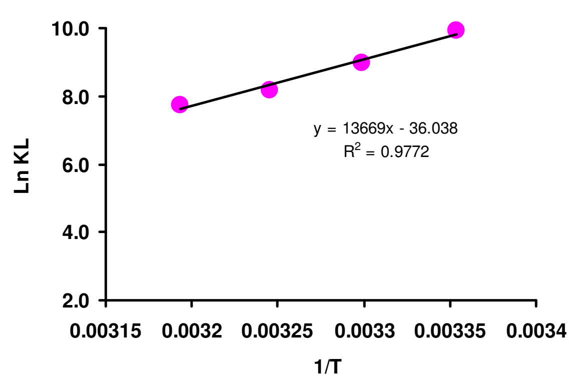

Fig. 5. van't Hoff plot of for the removal of Pb(II) by sulphuric acid-treated brown algae

Table 2. Thermodynamic constants for the recalculated thermodynamics parameters of Pb(II) biosorption by sulphuric acid-treated brown algae using the dimensionless K_C in comparison with the original results of Iddou et al. [1]. Values in parentheses indicate 95% confidence interval.

T (°C)	T (K)	K_C	ΔG° (kJ/mol)	ΔH° (kJ/mol)	ΔS° (J/(mol.K))
25	298.15	20220.90	-24.56 (-24.00 to -25.03)		
30	303.15	7879.26	-22.60 (-21.95 to -23.12)	-0.30	-299.47
35	308.15	3499.55	-20.90 (-18.87 to -22.01)	(-194.34 to 193.74)	(-553.60 to -45.35)
40	313.15	2322.27	-20.17 (-17.71 to -21.41)		

Result from original published work [1]

T (°C)	T(K)	K_L (L/mol)	ΔG° (kJ/mol)	ΔH° (kJ/mol)	ΔS° (J/(mol.K))
25	298.15	15.3	-15.09		
30	303.15	13.61	-14.62	-43.28	-94.59
35	308.15	10.79	-13.68		
40	313.15	7.56	-12.73		

Based on replot as suggested by Nguyen's method (**Fig. 5**), the parameters of $\Delta H°$ and $\Delta S°$ of adsorption thermodynamics from 298.15 to 313.15 K were recalculated (**Table 2**). Generally, the slope of van't Hoff plot defines the system either it is exothermic (negative slope) or endothermic (positive slope). There usually be an increase in the mobility of the adsorbate atoms or molecules as an increase in the number of active adsorption sites occurs with an increase in temperature due to the presence of heat or energy.

The standard adsorption enthalpy change was barely negative ($\Delta H°$ = -0.3 kJ/mol) that may initially indicate an exothermic reaction. The confidence interval value, however, span a large value that include a positive value even though the plot clearly indicates a positive slope. At this moment more data is needed to suggest whether the reaction is either exothermic or endothermic. As the linearization of the Arrhenius equation through the van't Hoff plot has its flaws compounded with the problem of low degree of freedom (with 4 data points), this might be the reason for the large confidence interval values observed in this study. In the future, a remedy to this problem has been suggested by Osmari et al [32].

A negative ΔS° value observed in this study normally indicates that the adsorption process exhibited a decrease in disorder at the solid/solution interface, which may reflect the existence of significant structural changes in adsorbent and adsorbate. On the other hand, a positive entropy changes ΔS° of adsorption normally suggest and increase in the randomness of the adsorbates after adsorption due to increase in temperature. Besides, the positive value of ΔS° might be because the adsorption occurs in two consecutive steps: desorption of the adsorbed water and adsorption of the adsorbate. In addition the negative value may mean that the biosorption process is enthalpy-governed rather than entropy-governed [18,33,34].

Very few works on biosorption report confidence 95% confidence interval values [12,13,35,36], but these values are for the parameter constant values of isotherm or kinetics and rarely for thermodynamic values. A 95% confidence interval is a range of values that it can be 95% certain to contain the true mean of the population. To confirm the accuracy and precision of the asymptotatic 95% confidence interval, Monte Carlo simulation may be carried out [37].

CONCLUSION

Based on the results obtained, it could be concluded that, thermodynamic parameters such as Gibb's free energy (ΔG), enthalpy (ΔH) and entropy (ΔS) have an influence on the adsorption process of Pb(II) ions removal using *Cystoseira stricta* biomass. The values of the thermodynamic parameters obtained with 95% confidence interval shows the probable range of phenomena that might explain the adsorption mechanism. The big range of values of the interval is a result of a few data points and confirm the accuracy and precision of the asymptotatic 95% confidence interval, Monte Carlo simulation is one of the ways this can be determined.

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