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Biosorption of Lead (II) Ions by Brown Algae: A Thermodynamic Study

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ABSTRACT

In the assessment of adsorption processes, thermodynamic experiments on the adsorption process play a critical role (both physical and chemical). The precision measurement of thermodynamic parameters relies on 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 alkali-treated brown algae *Cystoseira stricta* biomass at different temperatures with removal of outliers. 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 30, 35 and 40 ^{o}C of -18.74 (95% C.I., -19.49 to -17.98), -20.8 (95% C.I.,-21.49 to -20.11) and -21.82 (95% C.I.,-22.24 to -21.41), respectively, and occurred in an endothermic nature (ΔH= 0.31 kJ/mol (95% C.I., -107.90 to 108.52) with an increased in randomness (ΔS= 309.78 J/mol×K (95% C.I., 95% C.I., 275.92 to 343.64) kJ/mol). Resampling methods such as bootstrap, jacknife or Monte Carlo simulation may be carried out in the future to confirm the accuracy and precision of the asymptotatic 95% confidence interval, .

INTRODUCTION

Discharges of waste containing heavy metals have been a significant issue for years due to their harmful environmental effects [1,2]. Heavy metal pollution is one of the most significant contaminants present in the environment and is a huge concern in the world today. Environmental pollution caused by heavy metals, including chromium, manganese, iron, cobalt, nickel, copper, zinc, arsenic, cadmium, mercury, and lead, has been considered a major problem in recent years due to their toxicity and widespread occurrence [2]. Cleaning polluted water and soil is getting very difficult since heavy metals are non-biodegradable and have been in the environment for a long time. It is important to develop cost-effective technology that can easily be fish out from polluted water and soil. This can be achieved using modern treatment technologies [3]. Metals accumulate very quickly in living organisms, that is why their adverse effects can occur even at very low concentrations [4]. Pb can cause serious damage to the kidney, brain, liver, reproductive and nervous systems, as well as oxidative stress and compromised respiratory infections in humans [1,5]**.**

Biosorption is probably the most cost-effective form of remediation of toxins in water sources [6–13]. Algae has been historically used to sorb a variety of toxicants [8,14–24]. 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 [25].

There has been a recent interest in correcting the current problem of the K_L value from Langmuir modelling that in fact is a unit with dimension while thermodynamic calculation warrants that this value should be dimensionless. 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°, ΔH°, and ΔS°. The Langmuir constant in its dimensionless form [25] 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. [26] from **Fig. 8**. of Pb(II) sorption on alkali-treated brown algae at different temperatures modelled according to the Freundlich isotherms was processed using the software Webplotdigitizer 2.5 [27] 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 [25].

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 $\mathrm{gmol^{-1}}$. 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 ΔG°, ΔH° and ΔS° 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 (30, 35 and 40 °C). The presence of outliers were detected according to a previous methods [28] and then removed prior to modelling again with data points without the outliers.

RESULTS AND DISCUSSION

The absorption thermodynamics data from a published work [2] on the biosorption of Pb(II) by alkali-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 **3**.

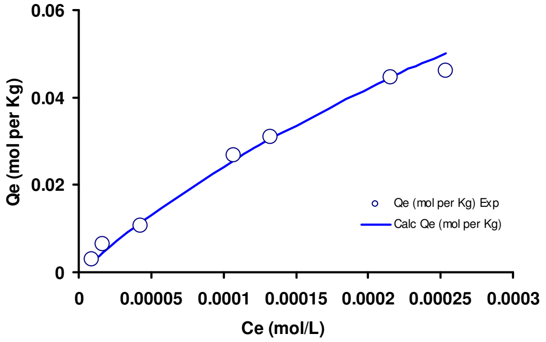

Fig. 1. Modelling of the adsorption of Pb(II) onto alkali-treated brown algae at 30 °C using the Langmuir model after the removal of outliers with recalculation of the equilibrium constant K_L, into a dimensionless form.

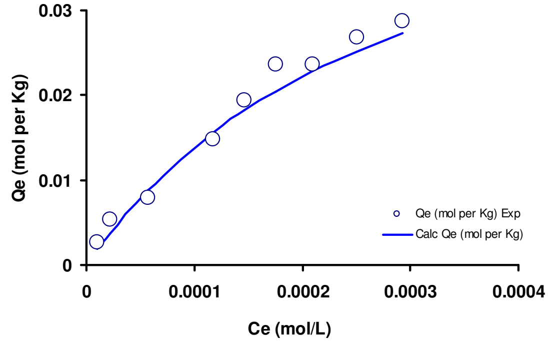

Fig. 2. Modelling of the adsorption of Pb(II) onto alkali-treated brown algae at 35 °C using the Langmuir model after the removal of outliers with recalculation of the equilibrium constant K_L, into a dimensionless form.

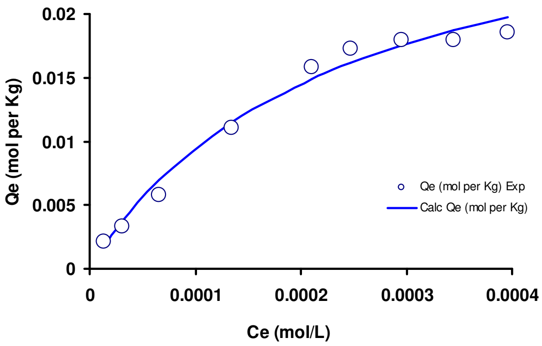

Fig. 3. Modelling of the adsorption of Pb(II) onto alkali-treated brown algae at 40 ºC using the Langmuir model after the removal of outliers with recalculation of the equilibrium constant K_L, into a dimensionless form.

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 [25]. 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 [29]. 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 part in the complete understanding of the mechanism of adsorption in aqueous solutions of organic and inorganic compounds. Thermodynamic adsorption is essential in the three experiments, taking into account the modes and processes of the adsorption mechanism under the temperature variation of the solution. Thermodynamics can essentially exhibit adsorption processes through a series of adsorptive balance experiments under varying temperatures and different initial adsorbate concentrations and fixed optimum conditions (pH of the solution, the adsorbent's particle size, ionic strength, and solid/liquid ratio) [25,30–39].

Thermodynamic criteria test the effectiveness and spontaneity of the process of adsorption. They are also important for the assessment of adsorbents (i.e. physisorption, ion exchange or chemisorption) [2,7]. The exact measurement of these thermodynamic parameters relies entirely on the constant balance 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 [25] 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. [26])	K_c (dimensionless). This study (± Std Error)
30	31.746	1698.31±207.63
35	29.325	3375.05±363.11
40	14.723	4385.05±269.99

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. 4**, 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].

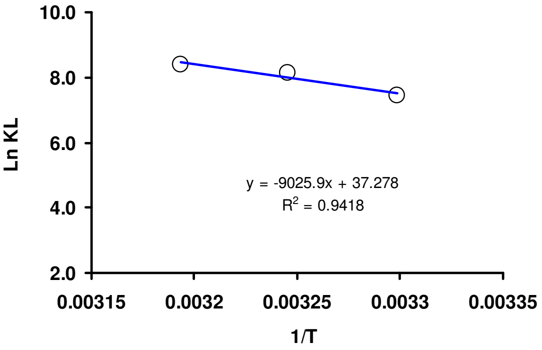

Fig. 4. van't Hoff plot of for the removal of Pb(II) by alkali-treated brown algae

Table 2. Thermodynamic constants for the recalculated thermodynamics parameters of Pb(II) biosorption by alkali-treated brown algae using the dimensionless K_C in comparison with the original results of Iddou et al. [40]. Values in parentheses indicate 95% confidence interval.

T (ºC)	T (K)	K_C	ΔG° (kJ/mol)	ΔH° (kJ/mol)	ΔS° (J/(mol.K))
30	303.15	1698.31	-18.74 (-19.49 to -17.98)	0.31	309.78
35	308.15	3375.05	-20.8 (-21.49 to -20.11)	(-107.90 to 108.52)	(275.92 to 343.64)
40	313.15	4385.05	-21.82 (-22.24 to -21.41)		

Result from original published work [40]

T (ºC)	T(K)	K_L (L/mol)	ΔG° (kJ/mol)	ΔH° (kJ/mol)	ΔS° (J/(mol.K))
30	303.15	31.746	-16.85	-49.83	-108.86
35	308.15	29.325	-15.56		
40	313.15	14.723	-14.67		

Based on replot as suggested by Nguyen's method (**Fig. 5**), the parameters of ΔH° and ΔS° of adsorption thermodynamics from 303.15 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 is a decrease in the mobility of the adsorbate atoms or molecules as a decrease in the number of active adsorption sites occurs with an increase in temperature.

The standard adsorption enthalpy change was barely positive (ΔH° = 0.31 kJ/mol) that may initially indicate an endothermic reaction. The confidence interval value, however, span a large value that include a negative value even though the plot clearly indicates a negative 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 3 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 [41].

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 [25,42,43].

Very few works on biosorption report confidence 95% confidence interval values [8,9,44,45], 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 [46].

CONCLUSION

Based on the results obtained, it could be concluded that the remodelling of the thermodynamic parameters such as Gibb's free energy (ΔG), enthalpy (ΔH) and entropy (ΔS) using the dimensionless Langmui's constant gave a largely different value and conclusion of the mechanism of adsorption 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, bootstrap and jacknife methods are several of the ways this can be determined.

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