

Equilibrium, kinetic and thermodynamic study of the biosorption of uranium onto *Cystoseria indica* algae

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Abstract

Biosorption equilibrium, kinetics and thermodynamics of binding of uranium ions to *Cystoseria indica* were studied in a batch system with respect to temperature and initial metal ion concentration. Algae biomass exhibited the highest uranium uptake capacity at 15 °C at an initial uranium ion concentration of 500 mg l⁻¹ and an initial pH of 4. Biosorption capacity increased from 198 to 233 mg g⁻¹ with an decrease in temperature from 45 to 15 °C at this initial uranium concentration. The Langmuir isotherm model were applied to experimental equilibrium data of uranium biosorption depending on temperature. Equilibrium data fitted very well to the Langmuir model *C. indica* algae in the studied concentration range of Uranium ions at all the temperatures studied. The saturation type kinetic model was applied to experimental data at different temperatures changing from 15 to 45 °C to describe the batch biosorption kinetics assuming that the external mass transfer limitations in the system can be neglected and biosorption is chemical sorption controlled. The activation energy of biosorption (E_A) was determined as -6.15 using the Arrhenius equation. Using the thermodynamic equilibrium coefficients obtained at different temperatures, the thermodynamic constants of biosorption (ΔG° , ΔH° and ΔS°) were also evaluated.

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1. Introduction

Due to the increasing environmental contamination caused by human industrial activity (mining industry, enrichment factories, etc.) the removal of radionuclides, including uranium, from water solutions is an actual problem. It is known that sorption can be effective to solve environmental tasks on purification of water solutions from radionuclides. There are many natural, organic and inorganic, adsorbents that have been used for uranium removal from water solutions [1–8]. These studies document that various biomasses from fungi, yeast, algae and unicellular bacteria are capable of uptake or binding of uranium greater than 15% of biomass and dry weight. A metal loading capacity of greater than 15% of biomass and dry weight has been defined as an economic threshold for practical applications of biosorption compared to alternative methods such as

traditional adsorption, ion exchange, chemical precipitation, solvent extraction, and reverse osmosis [8,9]. Pretreated *Cystoseria indica* show high metal loading capacity for the removal of uranium [10]. The pretreatment process enhances biomass surface ions. Since the biosorption of uranium is largely ion exchange process, the processed samples of the biomass have higher sorption capacity in comparison with untreated cells [10]. *C. indica*, brown algae are very abundant in Persian gulf on the coast of Qeshm, Iran. The mechanism of binding metal ions by inactivated algal biomass may depend on the species and ionic charges of metal ion, the algal organism, the chemical composition of the metal ion solution and other external environmental factors such as pH and temperature. Although the effects of pH, initial metal ion concentration, biomass concentration on biosorption of uranium by *C. indica* algae have been studied, information on the effect of temperature is still scanty. The present work extended the investigation of the biosorption by the *C. indica* algae biomass to the uranium removal. Kinetics data of this biosorption were analyzed using the saturation type adsorption kinetic model and kinetic constants were calculated depending

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Nomenclature

b	the Langmuir adsorption constant (l mg^{-1})
$C_{\text{ad,eq}}$	adsorbed metal ion concentration at equilibrium (mg l^{-1})
C_{eq}	residual metal ion concentration at equilibrium (mg l^{-1})
C_0	initial metal ion concentration (mg l^{-1})
E_A	activation energy of sorption (kJ mole^{-1})
k_1, k_0	rate constants of saturation type biosorption ($\text{l g}^{-1} \text{min}^{-1}; \text{l mg}^{-1}$)
K'_c	the apparent equilibrium constant of the biosorption system
K_c^0	the standard thermodynamic equilibrium constant
q	adsorbed metal ion quantity per gram of alga at any time (mg g^{-1})
q_{eq}	adsorbed metal ion quantity per gram of alga at equilibrium (mg g^{-1})
Q^0	the Langmuir adsorption constant (mg g^{-1})
r_{ad}	initial biosorption rate ($\text{mg g}^{-1} \text{min}^{-1}$)
R	gas constant ($8.314 \text{ J mole}^{-1} \text{ K}^{-1}$)
R^2	correlation coefficient
T	solution temperature ($^{\circ}\text{C}, \text{K}$)
X	alga concentration (g l^{-1})
ΔG°	the Gibbs free energy of biosorption (kJ mole^{-1})
ΔH°	enthalpy change of biosorption (kJ mole^{-1})
ΔS°	entropy change of biosorption ($\text{kJ mole}^{-1} \text{ K}^{-1}$)

on temperature. The activation energy of the biosorption process, which is an indicator of biosorption type and biosorption mechanism, was evaluated using these kinetic constants. Since the evaluation of the heat change of the biosorption process is very important for reactor design, the thermodynamics of the biosorption process was investigated.

2. Mathematical description

2.1. Equilibrium parameters of biosorption

Equilibrium data, commonly known as adsorption isotherms, are basic requirements for the design of adsorption systems. Several isotherm equations have been used for the equilibrium modeling of biosorption systems. Both of these have been applied for uranium biosorption by *C. indica* data, the Langmuir and the Freundlich models. The equilibrium data fitted very well to the Langmuir model [10]. So, in this study the classical the Langmuir adsorption model were used to describe the equilibrium between adsorbed metal ions on the algal cell (q_{eq}) and metal ions in solution (C_{eq}) at a constant temperature. The Langmuir equation which is valid for monolayer sorption onto a surface with a finite number of identical sites is given by Eq. (1).

$$q_{\text{eq}} = \frac{Q^0 b C_{\text{eq}}}{1 + b C_{\text{eq}}} \quad (1)$$

where Q^0 is the maximum amount of the metal ion per unit weight of alga to form a complete monolayer on the surface bound at high C_{eq} , and b is a constant related to the affinity of the binding sites. Q^0 represents a practical limiting adsorption capacity when the surface is fully covered with metal ions and assists in the comparison of adsorption performance, particularly in cases where the sorbent did not reach its full saturation in experiments. Q^0 and b can be determined from the linear plot of $1/q_{\text{eq}}$ versus $1/C_{\text{eq}}$ [11].

2.2. Kinetic parameters of biosorption

In order to investigate the mechanism of biosorption and potential rate controlling step such as mass transport and chemical reaction processes, kinetic models have been used to test experimental data. In this study it is assumed that the algal particles are spherical and only surface adsorption is occurring. When the experimental data is obtained in a well-agitated batch system the effect of external film diffusion on biosorption rate can be assumed not significant. It can be assumed that measured concentrations are equal to cell surface concentrations in this case [12].

The plot of q (the amount of adsorbed metal ion per gram of dried algae at any time) versus t (time) can be used to find the initial biosorption rate (r_{ad}) by differentiating the plot at $t=0$ as defined in Eq. (2).

$$\left(\frac{dq}{dt} \right)_{t=0} = r_{\text{ad}} \quad (2)$$

From experimental data, it was shown that the initial biosorption rate is proportional to the first power of the initial metal ion concentration at lower bulk metal ion concentrations and at higher metal ion concentrations, the rate becomes independent of initial metal ion concentration. Eq. (3) can be used to describe the rate of biosorption very accurately in both situations. This kind of rate equation is also defined as ‘saturation type’.

$$r_{\text{ad}} = \frac{k_1}{1 + k'_0 C_0} \quad (3)$$

where k_1 is the first-order rate constant of saturation type biosorption. The zero-order rate constant (k_0) is expressed as k_1/k'_0 . A straight line of $1/r_{\text{ad}}$ versus $1/C_0$ suggests the applicability of this kinetic model and k_1 and k_0 can be determined from the slope and intercept of the plot. This model predicts the adsorption behavior over the whole studied concentration range of metal ion and is in agreement with an adsorption mechanism being the rate controlling step [13].

The first-order rate constant of the biosorption reaction (k_1) is expressed as a function of temperature by the following Arrhenius type relationship:

$$k_1 = A_0 \exp\left(\frac{-E_A}{RT}\right) \quad (4)$$

where A_0 is the frequency factor, E_A the activation energy of sorption, R the gas constant and T is the solution temperature. When $\ln k_1$ is plotted versus $1/T$, a straight line with slope $-E_A/R$

is obtained. The magnitude of activation energy may give an idea about the type of sorption. Two main types of adsorption may occur, physical and chemical. In physical adsorption the equilibrium is rapidly attained and reversible, because the energy requirements are small. The activation energy for physical adsorption is usually no more than 4.2 kJ mole^{-1} , since the forces involved in physical adsorption are weak. Chemical adsorption is specific and involves forces much stronger than in physical adsorption. So the activation energy for chemical adsorption is of the same magnitude as the heat of chemical reactions. Two kinds of chemical adsorption are encountered, activated and, less frequently, nonactivated. Activated chemical adsorption means that the rate varies with temperature according to finite activation energy (between 8.4 and $83.7 \text{ kJ mole}^{-1}$) in the Arrhenius equation. In nonactivated chemical adsorption, chemisorption occurs very rapidly, suggesting the activation energy is near zero [13].

2.3. Thermodynamic parameters of biosorption

The biosorption process of metal ions can be summarized by the following reversible process which represents a heterogeneous equilibrium.

Metal ion in solution \leftrightarrow Metal ion–Biosorbent

The apparent equilibrium constant (K'_c) of the biosorption is defined as:

$$K'_c = \frac{C_{\text{ad,eq}}}{C_{\text{eq}}} \quad (5)$$

where $C_{\text{ad,eq}}$ is the concentration of metal ion on the adsorbent at equilibrium. In this case the activity should be used instead of concentration in order to obtain the standard thermodynamic equilibrium constant (K_c^0) of the biosorption system. If infinite dilution value of K'_c can be found by calculating the apparent equilibrium constant (K'_c) at different initial concentrations of metal ion and extrapolating to zero, this value will give K_c^0 . The K_c^0 value is used in the following equation to determine the Gibbs free energy of biosorption (ΔG°) at 25°C .

$$\Delta G^\circ = -RT \ln K_c^0 \quad (6)$$

The Gibbs free energy indicates the degree of spontaneity of the adsorption process and the higher negative value reflects a more energetically favorable adsorption. The equilibrium constant may be expressed in terms of enthalpy change of biosorption (ΔH°) and entropy change of biosorption (ΔS°) as a function of temperature. The relationship between the K_c^0 and temperature is given by the van't Hoff equation:

$$\ln K_c^0 = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (7)$$

ΔH° and ΔS° can be obtained from the slope and intercept of a van't Hoff plot of $\ln K_c^0$ versus $1/T$ [13,14].

3. Experimental

3.1. Preparation of algae and uranium solutions

C. indica, a brown alga, obtained from Persian Gulf on the coast of Qeshm, Iran was extensively washed with distilled water and sundried on the beach and in an oven at 50°C overnight. Dried biomass was ground in a laboratory blender and sorted by sieving using the standard test sieves. The batch of biomass with particle size 1.0 – 1.25 mm was selected for subsequent pretreatments. Pretreatment of the biomass was carried out as follows: a sample of 10 g of biomass was treated with 0.1 N HCl solution (1000 ml) for 12 h under slow stirring. After 12 h of contacting with acid, the biomass was rinsed with deionized water in the same volume many times until a stable wash solution pH was reached. The biomass was then dried in an oven at 50°C overnight. So, prepared biomass was stored for later uses.

Uranium solutions were prepared by diluting 1000 mg uranium/l stock solution which was obtained by dissolving a weighed quantity of uranium in nitrate salt form, $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, analytical grade. Diluted solutions were prepared at room temperature in distilled and deionized water. The range of concentrations of prepared solutions changed between 50 and 500 mg l^{-1} . The pH of each solution was adjusted to pH 4 with diluted or concentrated HCl and NaOH solutions before mixing the algae solution. This pH value was determined as the optimum pH value for the uranium biosorption [10].

3.2. Analysis of uranium concentration

Dissolved uranium concentrations in solution were assessed by an inductively coupled plasma atomic emission spectrophotometer (ICP-AES, Thermo Jarrel Ash, Model TraceScan). The ICP analyses were conducted at wavelength of 409.014 nm .

3.3. Sorption experiments

In order to determine the contact time required for the sorption equilibrium experiments, the sorption dynamics experiments were conducted first. On a shaker incubator 0.2 g of biomass was mixed with 100 ml of 500 mg U/l $\text{UO}_2(\text{NO}_3)_2$ solution at 150 rpm . A series of 0.5 ml samples of solution were removed from the vessel at predefined time intervals. After appropriate dilution, the samples were analyzed by the ICP-AES for metal concentrations. After the equilibrium time was determined, a series of uranium nitrate solution with different concentrations (100 ml) were mixed with 0.2 g biomass and the sorption equilibrium experiments were conducted in the same way. The suspensions were brought to the desired pH by adding 0.05 N HCl or NaOH during the sorption process and the volume of HCl or NaOH added was recorded. After 3 h of contact (according to the preliminary sorption dynamics tests), equilibrium was reached and the solution was filtered. The supernatant was diluted for uranium concentration analyses by the ICP-AES.

The amount of uranium ions adsorbed per unit of empty sorbent was obtained by using the following expression:

$$q = (C_i - C_e) \frac{V}{M} \quad (8)$$

where q is the amount of uranium adsorbed onto the unit mass of the adsorbent (mg/g), C_i and C_e the concentrations of the metal ions before and after biosorption (mg/l), V the volume of the aqueous phase (l), and M is the amount of the adsorbent (g).

All sorption experiments were carried out in triplicate. Values used in calculations were the arithmetic averages of the experimental data.

4. Results and discussion

4.1. Effect of contact time and temperature on uranium biosorption

The equilibrium uptake of uranium ions to the dried *C. indica* brown algae was affected by temperature and increased with decreasing temperature as shown in Table 1. Uranium biosorption was exothermic thus the extent of adsorption decreased with increasing temperature. The sorption of uranium ions by dried *C. indica* may involve not only physical but also chemical sorption. Fig. 1 shows the biosorption kinetics of uranium ion at 15, 25, 35 and 45 °C by plotting the uranium uptake capacity, q , versus time. The biosorption capacity increased with increasing contact time and a larger amount of uranium was removed by dried *C. indica* in the first 20 min of contact time. Equilibrium was established in 120–150 h at the end of a rapid biosorption for all the temperatures studied. The metal uptake versus time curves at different temperatures is single, smooth and continuous leading to saturation, suggesting possible monolayer coverage

Table 1
The effect of initial uranium concentration and temperature on the initial biosorption rate and equilibrium uptake capacity of *C. indica*

T (°C)	C_0 (mg/l)	q_{eq} (mg g ⁻¹)	r_{ad} (mg g ⁻¹ min ⁻¹)
15	50	24	2.43
	200	96	7.14
	350	163	11.41
	500	233	17.16
25	50	24	2.25
	200	95	6.9
	350	158	11.08
	500	216	16.62
35	50	24	2.1
	200	94	6.69
	350	152	10.78
	500	207	16.31
45	50	24	1.96
	200	93	6.49
	350	147	10.48
	500	198	15.16

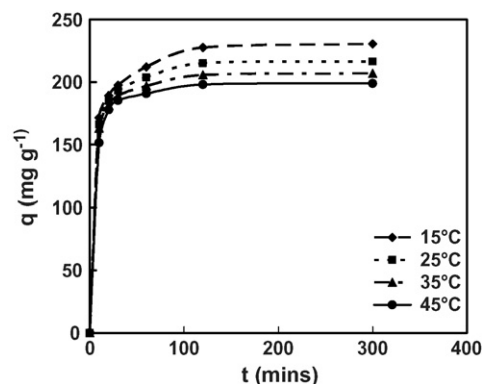


Fig. 1. The biosorption curves of uranium ions obtained at 500 mg l⁻¹ initial uranium concentration at different temperatures (initial pH: 4; X: 2.0 g l⁻¹; agitation rate: 150 rpm).

of uranium ions on the surface of the biosorbent. The observed rapid kinetics has also significant practical importance as it will facilitate the scale-up of the process to smaller reactor volumes ensuring efficiency and economy.

4.2. Effect of initial uranium concentration on temperature-dependent biosorption

The initial concentration provides an important driving force to overcome all mass transfer resistance of uranium between the aqueous and solid phases. Hence, a higher initial concentration of uranium will increase the adsorption rate. Such an effect was clearly demonstrated in Table 1 with respect to temperature. Since cells offer a finite number of surface binding sites, uptake showed saturation at higher metal ion concentrations. The temperature also influenced both the initial biosorption rate and equilibrium metal uptake. With the change in temperature from 15 to 45 °C, both the uptake capacity and biosorption rate decreased.

4.3. Determination of equilibrium model constants

Analysis of the equilibrium data is important to develop an equation which accurately represents the results and which could be used for design purposes. For each isotherm, initial uranium concentrations were varied while the dry cell weight in each sample was held constant. The linearized Langmuir adsorption isotherm of uranium ions obtained at the temperatures of 15, 25, 35 and 45 °C are given in Fig. 2. The Langmuir adsorption constants evaluated from the isotherms at different temperatures and their correlation coefficients are also presented in Table 2. High regression correlation coefficients (>0.985) were found, suggesting that this model are very suitable for describing the biosorption equilibrium of uranium by the algal cells in the studied concentration range at all the temperatures studied. The applicability of the Langmuir isotherm model to the uranium-dried algae system implies that monolayer biosorption conditions exit under the experimental conditions used. The monocomponent Langmuir constant, Q^0 , represents the monolayer saturation at equilibrium. The other monocompo-

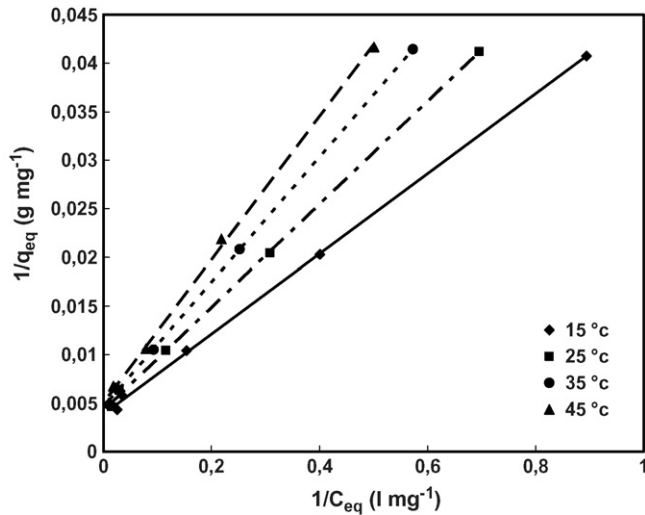


Fig. 2. The linearized Langmuir adsorption isotherms of uranium biosorption by *C. indica*.

Langmuir constant b , corresponds to the concentration at which a uranium ion amount of $Q^0/2$ is bound and indicates the affinity for the binding of uranium ions. A high b value indicates a high affinity. Values of Q^0 and b for different temperatures were calculated from the Langmuir plots in Fig. 2 and the results are also tabulated in Table 2. The maximum capacity Q^0 determined from the Langmuir isotherm defines the total capacity of the biosorbent for uranium. The adsorption capacity of biosorbent also decreased on increasing the temperature. The value of Q^0 obtained at 15 °C (i.e. maximum uptake and equal to 256 mg g⁻¹) appears to be higher in comparison with the uptake obtained at the other temperatures. A higher value of b also implied strong bonding of uranium to the dried *C. indica* at this temperature.

4.4. Determination of kinetic constants

In order to analyze the biosorption kinetics of uranium ions, the saturation type kinetic model was applied to the experimental data. Fig. 3 shows the plots of linearized form of the saturation type kinetic model at all temperatures studied. The rate constants k_1 and k_0 values determined from Fig. 3 at 15, 25, 35 and 45 °C are compared with the correlation coefficients in Table 3. The correlation coefficients obtained are greater than 0.996 for all temperatures studied. The saturation type rate constants also decreased with increasing temperatures. The change of rate constant due to temperature also show the biosorption is sorption rate controlled. The obtained kinetic parameters can be used for

Table 2
The adsorption constants obtained from the Langmuir adsorption isotherms of uranium by *C. indica* at different temperatures

T (°C)	Q^0 (mg g ⁻¹)	b (l mg ⁻¹)	R^2
15	256	0.0944	0.994
25	233	0.0809	0.995
35	217	0.0714	0.994
45	200	0.0677	0.985

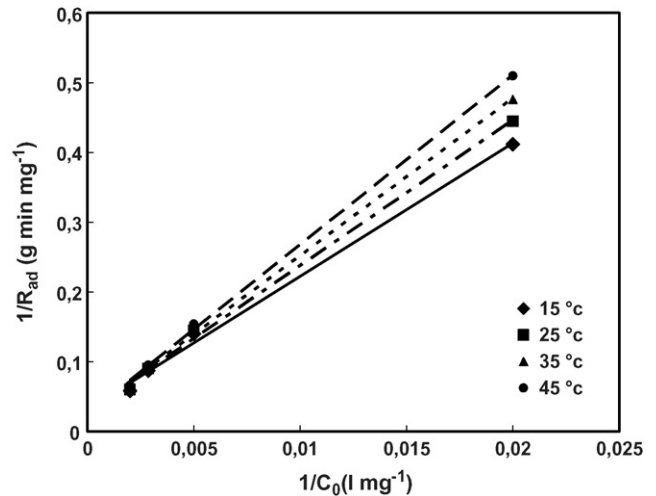


Fig. 3. The linearized saturation type kinetic equation of uranium biosorption by *C. indica* plots obtained at different temperatures.

Table 3

The comparison of the saturation type kinetic rate constants obtained at different temperatures

T (°C)	k_1 (l g ⁻¹ min ⁻¹)	k_0 (l mg ⁻¹)	R^2
15	0.0524	0.0017	0.996
25	0.0479	0.0014	0.997
35	0.0443	0.0012	0.998
45	0.0411	0.001	0.999

bioreactor design. It may be suitable to apply such simple kinetic models to a well-agitated batch biosorption system consisting of a free cell suspension neglecting external film diffusion.

Fig. 4 shows the corresponding linear plot of $\ln k_1$ against $1/T$ with a high correlation coefficient of 0.9998. The activation energy for the biosorption system of uranium onto *C. indica* was -6.15 kJ mole⁻¹ derived from the slope of this plot. These findings showed that uranium biosorption process by *C. indica* is exothermic and involves both physical and chemical sorption.

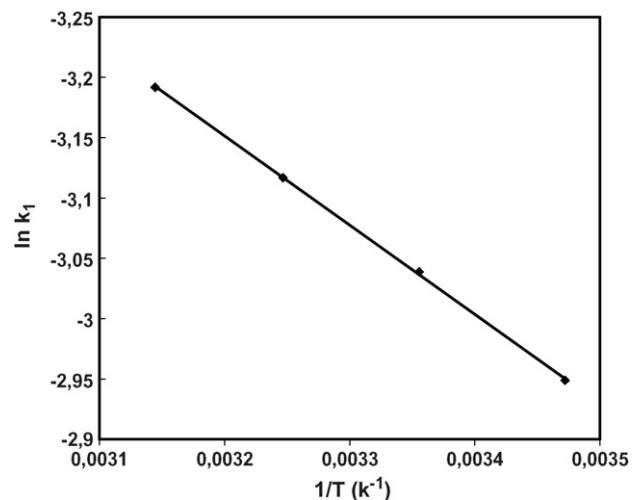


Fig. 4. The linearized Arrhenius relationship plot of uranium biosorption by *C. indica*.

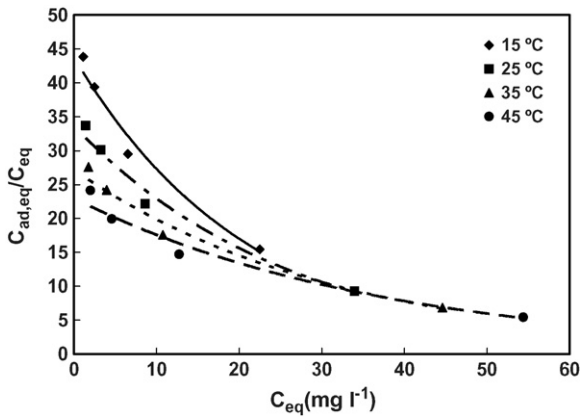
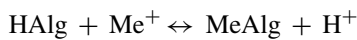


Fig. 5. The apparent equilibrium constant vs. C_{eq} in order to obtain the standard thermodynamic equilibrium constant.

Numerous chemical groups have been suggested to contribute to biosorption metal binding by algae. In brown alga biomass, alginate in the cell wall is the main component responsible for the chemical sorption. It is present in a gel form in the cell wall which appears very porous and easily permeable to small ionic species [15]. Ion exchange has been confirmed to be highly involved to a large degree in the metal sequestering by algal biomass as it is shown in the following reaction:



4.5. Determination of thermodynamic constants of biosorption

The K_c^0 value evaluated from the $C_{ad,eq}/C_{eq}$ versus C_{eq} plot (Fig. 5) was used to find the ΔG° value. The standard Gibbs free energy for the biosorption process was obtained at 25 °C as $-8.7 \text{ kJ mole}^{-1}$ using Eq. (6). The negative value of ΔG° is due to the fact that the biosorption process is spontaneous with high affinity of uranium to dried algae.

The standard enthalpy and entropy changes of biosorption determined from the $\ln K_c^0$ versus $1/T$ plot (Fig. 6) were $-16.39 \text{ kJ mole}^{-1}$ and $-0.026 \text{ kJ mole}^{-1} \text{ K}^{-1}$, respectively with a correlation coefficient of 0.995. The negative value

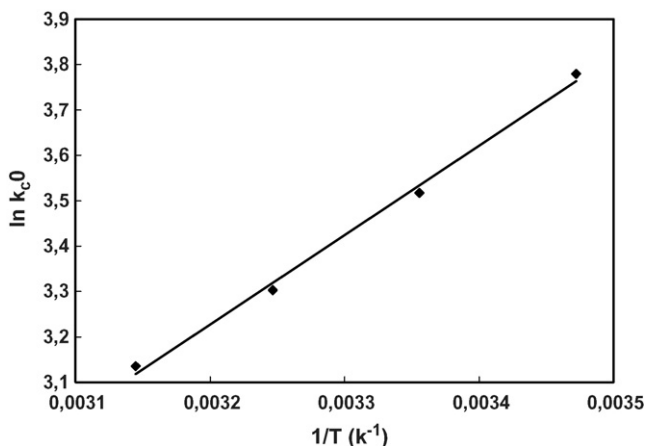


Fig. 6. The van't Hoff equation plot for obtaining ΔH° and ΔS° .

of ΔH° suggests the exothermic nature of biosorption. The negative value of ΔS° confirms the decreased randomness at the solid-solution interface during biosorption and reversibility of uranium biosorption.

5. Conclusion

The results obtained show that temperature and initial metal ion concentration highly affected the uptake capacity of the biosorbent. Biosorption increased on decreasing temperature up to 15 °C and increasing the uranium concentration up to 500 mg l^{-1} . The Langmuir adsorption model were used for the mathematical description of the biosorption equilibrium of uranium ions to dried *C. indica* depending on temperature. Results showed that the adsorption equilibrium data fitted very well to the Langmuir model in the studied concentration range at all the temperatures studied. It was seen that the isotherm constants increased with decreasing temperature. The suitability of the saturation type kinetic model for the sorption of uranium ions onto biomass was also discussed assuming no effect of mass transfer on the biosorption rate. The results calculated due to this model were found to be in good agreement with the experimental results. Using the first-order kinetic constant of saturation type kinetic model increased with decreasing temperature, the activation energy of biosorption was determined as $-6.15 \text{ kJ mole}^{-1}$ showing that the biosorption process is exothermic. Thermodynamic constants were also evaluated using equilibrium constants changing with temperature. The negative value of ΔG° indicated the spontaneity and the negative values of ΔH° and ΔS° showed the exothermic nature and reversibility of uranium biosorption, respectively. We believe that application of biosorption by the brown alga in purification of wastewater for the removal of uranium ions from industrial wastewaters can be suitable for large-scale exploitation by using these kinetic parameters. More studies are needed to optimize the system from the regeneration point of view and economic variances.

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