

# Biosorption of Radioactive Thorium by *Sargassum filipendula*

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## Abstract

In the present work, the biosorption of radioactive thorium was investigated using a dry biomass of *Sargassum filipendula* as the biosorbent material. Radioactive solutions containing between 2.0 and 500.0 µg thorium were tested by biosorption with *S. filipendula*, yielding uptake capacities from 20 to 100%, depending on the concentration of the solution. Kinetic studies indicated that equilibrium between the thorium solution and the solid fraction was achieved after three hours of contact and that a second-order model could express the equilibrium kinetics. In order to investigate the maximum biosorption capacity of the biomass an isotherm was done, based on the experimental data, which revealed the maximum uptake capacity to be 2.59 µmol thorium/g biomass. The experimental data fitted well to a Langmuir model, which provided a good correlation between the experimental and predicted thorium uptake values.

**Index Entries:** *Sargassum filipendula*; thorium; wastewater; biosorption; kinetic models.

## Introduction

There are increased concerns worldwide about the inappropriate disposal of heavy metals into aqueous streams, especially when these wastewaters include radioactive tracers. Many types of soils and ores present

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natural radioactivity, which cannot be underestimated even though it is much lower than man-made emissions of radioactive compounds. Aside from these emission sources, the routine procedures adopted for chemical, biochemical and biomedical analyses can also generate liquid effluents that contain high levels of heavy metals and radioactive elements (1–3).

In Brazil, the institution in charge of monitoring the environmental radioactive impacts arising from the operation of nuclear power plants is the Instituto de Radioproteção e Dosimetria (IRD). It is also responsible for monitoring mining operations and ore dressing, where natural radionuclides are present, as well as any other activity involving radiochemical emissions that could endanger the population.

During its work, the IRD carries out a wide variety of analytical procedures. These include constant monitoring of environmental samples, such as soils, foods, plants, and waters using radiochemical/radiometric techniques. Some of these activities include monitoring natural radioactivity—the main purpose of this project is to evaluate natural background levels of radioactivity, considering the different demographics and climatic changes in the country; natural radiation augmented by technology—an evaluation of the environmental impact of activities capable of increasing people's exposure to natural radioactivity, such as nonradioactive mining and the use of phosphogypsum; and the analysis of environmental samples from nuclear power plants and research projects with a view to improving control protocols and understanding the transport mechanisms of radionuclides in a variety of environmental media.

All of these activities can produce high quantities of inorganic pollutants in laboratories, especially from the radiochemical analyses performed on samples from the environment. For instance, thorium (Th) is determined through arsenazene III; uranium (U) is determined through fluorimetry; radioactive lead ( $\text{Pb}^{210}$ ) is determined by co-precipitation followed by  $\beta$  emission measurements; radioactive strontium ( $\text{Sr}^{90}$ ) is determined through ion-exchange and  $\beta$  emission; and radioactive radium ( $\text{Ra}^{226}$ ) is determined by co-precipitation and  $\alpha$  emission, among other elements.

The radiochemical techniques used to determine these elements in environmental samples, such as water, vegetables, sediments, and soils, produce laboratory wastewaters containing high levels of chromium and barium, as well as radioactive tracers. Aside from these radioactive tracers, the concentrations of U, Th,  $\text{Sr}^{90}$ ,  $\text{Ra}^{226}$ ,  $\text{Ra}^{228}$ ,  $\text{Pb}^{210}$ ,  $\text{Pu}^{238}$ ,  $\text{Pu}^{239}$ ,  $\text{Po}^{210}$ , and  $\text{H}^3$  produced in regular laboratory routines require them to be immobilized before such wastewaters can be discharged into the environment.

The main aim of this work was to make a preliminary evaluation of the use of biosorption for treating wastewaters containing radiochemical elements using brown seaweeds of the *Sargassum filipendula* species. *S. filipendula* was selected as the biosorbent based on previous experiments for cadmium uptake using seaweeds of the *Sargassum*, *Padina*, *Codium*, *Colpomenia*, *Ulva*, and *Gelidium* genera. Further studies using the same

seaweeds for zinc, manganese, copper, and lead indicated the superior performance of the *Sargassum* biomass as compared to the other genera. This is potentially important because Brazil is totally dependent on imports of ion-exchange resins for this purpose and also because of the continuous generation of effluents containing radioactive thorium and stable heavy metals from the analysis of environmental samples.

## Materials and Methods

### Seaweed

The biomass used in the present work was the brown seaweed *S. filipendula* (Chromophyta) collected from the northeastern coast of Brazil. Intact *S. filipendula* was collected from the sea, sampled, washed thoroughly with distilled water to remove particulate material from the surface and oven-dried at 55°C for 24 h. From a bulk sample harvested from the sea, 1 kg biomass was subsampled for use in the experiments.

### Preparation and Quantification of Thorium Solutions

The thorium solutions used in the biosorption batch tests were prepared from a concentrated thorium nitrate solution dissolved in 1 M nitric acid solution until they reached a final volume of 50 mL.

Ten milliliters of each solution were transferred to extraction tubes. In order to extract thorium to the organic phase, 3 mL of a 0.1 M TOPO (Tri-n octyl phosphyn) in MIBC (Methyl isobutyl ketone) solution was used, followed by agitation for 3 min. Arsenaze III was added to the thorium in the organic phase for the spectrophotometry to be carried out. For the spectrophotometry, 1 mL of the organic phase was used together with 1 mL of an alcoholic solution of oxalic acid (4%), which led to the formation of compounds with the rare earth elements, which are usually present in thorium solutions.

The medium was acidified with 2 mL concentrated HCl. Green colored complexes then developed, and the results were obtained by spectrophotometry at 660 nm. Pure ethanol was added up to 9 mL and the reaction flasks were left to stand for 15 min as a result of the exothermic nature of the reaction.

A control flask was prepared, containing oxalic acid, TOPO in MIBIC, ethanol, and hydrochloric acid. When necessary, this solution was used to dilute the different samples so they would fit into a specific range of the spectrophotometric curves. The laboratory flasks were washed with 1 M HCl, then rinsed three times to ensure total decontamination. After this, the flasks were placed in a solution of Extran Detergent and deionized water and washed to ensure that they were totally clean. The different solutions containing thorium were placed in separate flasks for further classification of the radioactive waste.

### Preliminary Tests of Thorium Biosorption

Preliminary tests were performed using thorium solutions containing low, intermediate, and high thorium masses with the purpose of evaluating the potential use of *S. filipendula* for the biosorption of thorium. The quantities of thorium used in the solutions in these preliminary tests were as follows:

low thorium masses: 2.0, 3.0, 4.0, and 5.0  $\mu\text{g}$ ;  
 intermediate thorium masses: 6.0, 7.0, 10.0, and 20.0  $\mu\text{g}$ ; and  
 high thorium masses: 30.0, 40.0, 50.0, and 500.0  $\mu\text{g}$ .

The thorium solutions were prepared and 0.1 g *S. filipendula* was added. After this, the samples were placed in a rotary shaker for 24 h at  $25 \pm 1^\circ\text{C}$ . They were then filtered to separate the biomass from the bulk solution using Millipore filters. The filtered solutions were used to quantify equilibrium thorium concentrations, as previously described.

The results reported are the means of three experiments, and the mean standard deviations are included.

### Kinetic Study

A kinetic study was performed for the thorium biosorption process by *S. filipendula* in order to ascertain when thorium reaches equilibrium with the solid biomass phase. Fifty milliliters of a thorium solution containing Th-232 at 1 mg/L (50  $\mu\text{g}$  Th-232) and pH 4.0 were placed in several Erlenmeyer flasks. Biomass (0.1 g) was added to each flask, and these were all placed in a rotary shaker at  $25 \pm 1^\circ\text{C}$ , at 150 rpm. Each Erlenmeyer flask was removed from the shaker after a different period of time, which ranged from 1 min to 5 h. When removed from the shaker, each solution was immediately filtered so the biomass could be retained. The same experiment was performed three times and the mean results are reported. The mean standard deviations are included.

### Kinetic Modeling

The data obtained from the kinetic study were employed for the kinetic modeling of the biosorption of thorium by *S. filipendula*. Two different kinetic models were used to adjust the experimental data. A pseudo first-order Lagergren model (Eq. 1) was first considered:

$$\frac{dQ}{dt} = k_{1,ads} (Q_e - Q) \quad (1)$$

in which  $Q_e$  (mg/g) and  $Q$  (mg/g) are the amounts of adsorbed metal ions in the biosorbent at the equilibrium and at any time  $t$ , respectively, whereas  $k_{1,ads}$  is the Lagergren rate constant of first-order biosorption. By integrating Eq. 1 between the limits,  $t = 0$  to  $t = t$  and  $Q = 0$  to  $Q = Q_e$ , it becomes Eq. 2:

$$\log(Q_e - Q) = \log Q_e - (k_{1,ads} / 2.303)t \quad (2)$$

The linear plots of  $\log(Q_e - Q)$  against  $t$  indicate the applicability of this kinetic model. The  $Q_e$  value must be pre-estimated by extrapolating the experimental data to  $t \rightarrow \infty$ .

The second-order model is based on the assumption that the rate of occupation of adsorption sites is proportional to the square of the number of unoccupied sites (Eq. 3):

$$\frac{dQ}{dt} = k_{2,ads} (Q_e - Q)^2 \quad (3)$$

in which  $k_{2,ads}$  is the rate constant of second-order biosorption (g/mg.min). By integrating Eq. 3 for boundary conditions  $t = 0$  to  $t = t$  and  $q = 0$  to  $Q = Q_e$ , and then linearizing Eq. 4:

$$\frac{t}{Q} = \left( \frac{1}{k_{2,ads}} \frac{Q_e^2}{Q_e^2} \right) + \left( \frac{1}{Q_e} \right) t \quad (4)$$

$Q_e$  and  $k_{2,ads}$  can be calculated from the slope and the interception of the  $t/Q$  plot against  $t$ . It is important to note that although an estimate of the experimental value for  $Q_e$  was obtained, it was not necessary for this model to be applied. By plotting  $t/Q$  against  $t$ , a straight line could be obtained for all the biomass concentrations under study, by which the second-order rate constant ( $k_{2,ads}$ ) and  $Q_e$  values could be determined.

### Batch Biosorption Isotherm

In these experiments, 50-mL portions of thorium solution, containing between 2.38 and 190.67  $\mu\text{g}$  thorium at pH 4.0, plus *S. filipendula* at 2.0 g/L, were incubated in a rotary shaker for 3 h at  $25 \pm 1^\circ\text{C}$ . These experiments were performed twice, and the mean results are reported. After they had been incubated in the shaker, the samples were filtered and the thorium was quantified according to the previously described method.

The thorium uptake ( $Q$ ) was calculated by the concentration difference method. The initial concentration  $C_0$  (mg/L) and the metal concentration at any time,  $C_t$  (mg/L) were determined. The metal uptake  $Q$  (mg thorium/g *S. filipendula*) was calculated from the mass balance (Eq. 5):

$$Q = (C_0 - C_t) \frac{V}{1000w} \quad (5)$$

in which  $V$  is the solution volume (mL) and  $w$  is the sorbent mass (g). The results reported constitute the mean results from three identical experiments. Mean standard deviations are included.

### Modeling Based on Langmuir and Freundlich Equations

The experimental data were used to check the suitability of classical adsorption models for explaining the uptake process involved. The Langmuir

and Freundlich equations are the most widely used in biosorption (Eqs. 1 and 2). The Langmuir model is as follows (Eq. 6):

$$Q_e = \frac{Q_0 K_L C_e}{1 + K_L C_e} \quad (6)$$

in which  $Q_0$  (mg/g) is the maximum amount of metal ion per unit weight of algae to form a complete monolayer on the surface.  $C_e$  is the equilibrium concentration and  $K_L$  is the equilibrium adsorption constant, related to the affinity of the binding sites.  $Q_0$  is the constant that represents the maximum adsorption capacity (Eq. 3). Parameters can be determined from a linearized form of Eq. 6, represented by Eq. 7:

$$\frac{C_e}{Q_e} = \frac{1}{Q_0 K_L} + \left(\frac{1}{Q_0}\right) C_e \quad (7)$$

By plotting  $(C_e/Q_e)$  against  $C_e$ , a straight line of a  $1/Q_0$  slope and a  $1/(Q_0 K_L)$  intercept are attained. The Freundlich equation is given by Eq. 8:

$$Q_e = K_F (C_e)^{1/n} \quad (8)$$

in which  $K_F$  and  $n$  are the Freundlich constants, indicating the adsorption capacity and adsorption intensity, respectively. The derivation of  $K_F$  and  $1/n$ , can be linearized in logarithmic form Eq. 9:

$$\log Q_e = \log K_F + \left(\frac{1}{n}\right) \log C_e \quad (9)$$

## Results and Discussion

### *Preliminary Tests of Thorium Biosorption*

The preliminary thorium biosorption tests were performed for solutions with a low, intermediate, and high concentration of thorium. The results for the biosorption by *S. filipendula* are presented in Fig. 1 as the percentage of the radioactive element biosorbed.

The results clearly indicate a decrease in the biosorption of thorium by *S. filipendula* as the concentration of the radionuclide increased in low and high thorium mass solutions. This is predictable behaviour that can be explained by the mass transfer mechanisms from liquid phase (thorium solution) to solid phase (biomass surface), as the same amount of biomass was used for increasing concentrations of the radioactive tracer. For instance, at 500  $\mu\text{g/mL}$  (2.05  $\mu\text{mol/L}$ ), there was 25.13% thorium biosorption, whereas for a less concentrated solution (50  $\mu\text{g/mL}$ , or 0.205  $\mu\text{mol/L}$ ), there was 60.95% thorium biosorption.

However, for the highly diluted samples (containing 2.0 to 5.0  $\mu\text{g}$  thorium), biosorption did not increase as significantly if compared to the intermediate concentration solutions. This can be explained by the diffusion of the radioactive tracer toward the surface of the biomass being hampered by the high dispersion of the element in the solution. For the intermediate

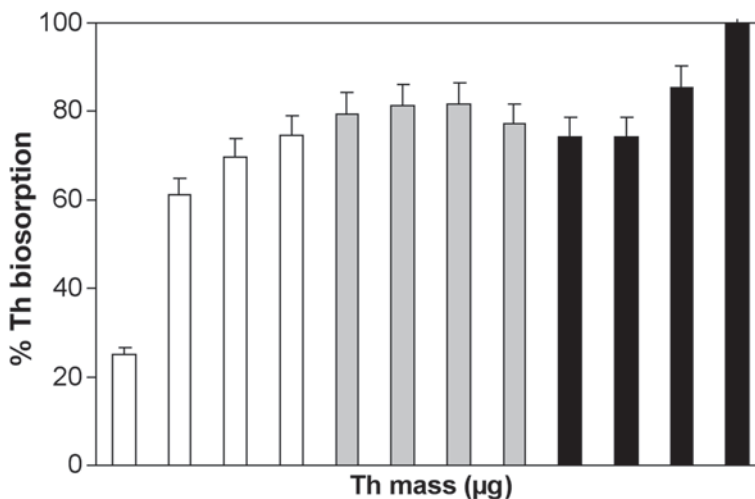


Fig. 1. Thorium biosorption by dry biomass of *Sargassum filipendula* using diluted thorium solutions (black bars), intermediate thorium solutions (gray bars), and concentrated thorium solutions (white bars).

thorium mass solutions, the thorium uptake did not decrease as the concentration of the element increased.

Irrespective of the fact that mass transfer mechanisms affected the biosorption in very diluted solutions, the thorium uptake was nonetheless good in the different very diluted solutions, indicating the viability of using *S. filipendula* brown seaweed for biosorption purposes. This potential biotreatment of weak, intermediate, and concentrated thorium solutions is highly attractive as it points to the chance of treating large volumes of solution, even if they contain low concentrations of thorium.

Similar behavior was observed by Aslani et al. (4) and Volesky and Yang (5), who mentioned the difficulty of treating high volumes of solutions contaminated with low concentrations of heavy metals using conventional technologies, such as chemical precipitation.

### Kinetic Study

As stated before, kinetics was investigated in order to ascertain the minimum contact time between the biomass and the thorium solution for equilibrium to be reached. The results are presented in Fig. 2.

The results clearly indicate that thorium biosorption is a slow process if compared to the conventional biosorption of other heavy metals. According to the results, the minimum time required to achieve equilibrium between *S. filipendula* and thorium in solution is 180 min, under the experimental conditions established.

In his study of the biosorption kinetics of  $\text{Cr}^{3+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Cu}^{2+}$  by the cyanobacterium *Spirulina* sp., Chojnacka (6) reached equilibrium between the biomass and the metals in approx 10 min. When Diniz and Volesky (7)



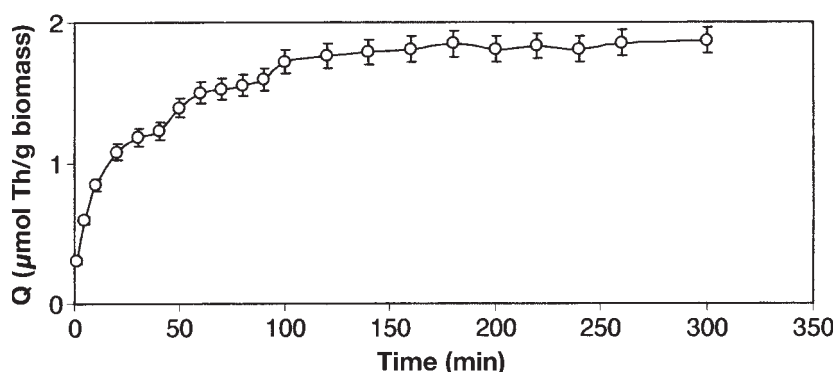


Fig. 2. Kinetic evaluation of thorium biosorption by dry biomass of *Sargassum filipendula*.

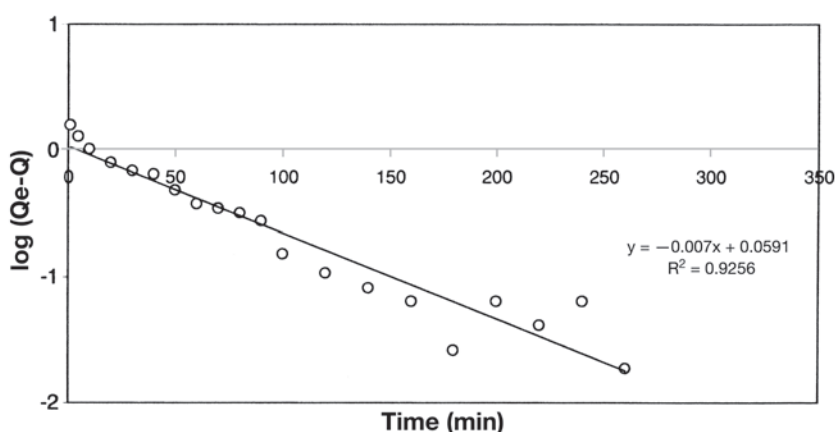


Fig. 3. Kinetic modeling of thorium biosorption by *Sargassum filipendula*—Lagergren pseudo first-order model.

studied the biosorption of La, Eu, and Yb by *Sargassum polycystum*, they reached equilibrium between the biomass and metal solution in 10 to 30 min, which points to the rapid nature of biosorption.

It should be noted that a comparison was made between a cyanobacterial strain and *S. filipendula*, and between thorium biosorption and rare-earth elements by *S. polycystum*, because no papers were found in the literature reporting on thorium and *Sargassum* biomass. However, we are aware that comparisons of this kind are subject to a series of additional considerations, such as the different compositions of the cell walls and different diffusional characteristics of the elements involved in the uptake.

When Volesky and Yang (5) used *Sargassum fluitans* as a biosorbent for uranium, they found that 3 h were needed to reach equilibrium. In recovering uranium by using oak tree biomass, Psareva and Zakutetskyy (8) observed that 2-h contact was required for equilibrium to be reached. These results obtained for radioactive uranium are in accordance with the present



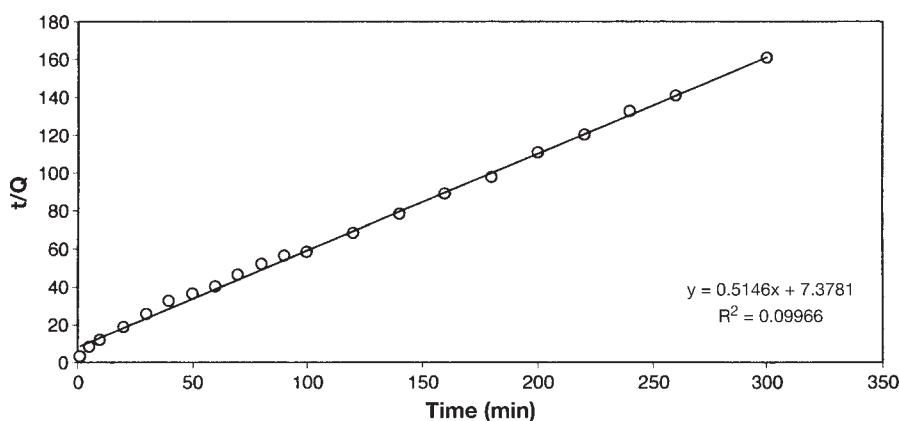


Fig. 4. Kinetic modeling of thorium biosorption by *Sargassum filipendula*—second-order model.

Table 1  
Kinetic Parameters From Pseudo First-Order and Second-Order Kinetics:  
Thorium Biosorption by *Sargassum filipendula*

Kinetic model	$k_{ads}$	$Q_e$ ( $\mu\text{mol/g}$ ) (calculated)	$Q_e$ ( $\mu\text{mol/g}$ ) (experimental)	$R^2$
Pseudo-first-order	0.016	1.146	1.870	0.9256
Second-order	0.036	1.943	1.870	0.9966

work, indicating that actinide radioactive tracers present different behavior in solution from conventional heavy metal elements.

### Kinetic Modeling

In order to check the suitability of kinetic models to explain the results obtained in the present work, a pseudo-first-order and a second-order model were selected. The results from the adjustment of data to the pseudo-first-order model are presented in Fig. 3, whereas those of the second-order model are presented in Fig. 4.

A preliminary evaluation of the results indicates that both models could be used to fit the experimentally obtained data. However, in order to check the relative suitability of the models, a comparison was made between the kinetic parameters, as shown in Table 1.

The results from Table 1 are in line with previous analyses of the models' suitability in explaining the experimental results. The second-order model best fits the experimental data, primarily because of the much higher correlation coefficient ( $R^2$ ) obtained. The results for the theoretical  $Q_e$  values also confirm the choice of the second-order model. When the theoretical  $Q_e$  value predicted by the pseudo first-order Lagergren (1.146) is compared to the experimental value (1.870), the difference is quite high, whereas the

theoretical value of 1.943 for  $Q_e$  yielded by the second-order model is close to the experimental value obtained for this parameter, which corroborates the choice of the second-order model to fit the experimental data. This is an indication that the rate of occupation of biosorption sites is proportional to the square of the number of unoccupied sites on the surface of the biomass.

As mentioned previously, no studies have been encountered about the biosorption of thorium by brown seaweeds for the purposes of comparison with the present results. However, the literature does present some results about the kinetic constants obtained during the uptake of radioactive tracers by microbial strains. Given the absence of any more suitable parameters to compare with our results, a comparison with microbial cells will be performed, even though the diffusion properties of stable or radioactive elements toward microbial cells is much more complex than the mass transfer mechanisms present in this work. For instance, the heterogeneous structures in multiple layers at the surface of microbial cells make the diffusion processes different from those predicted by the models selected for the present study.

When studying the adsorption of thorium and uranium by the bacterium *Micrococcus luteus* Nakajima and Tsuruta (9) observed  $Q_e$  values of 360  $\mu\text{mol/g}$  cells for uranium and 163  $\mu\text{mol/g}$  cells for thorium. They also found adsorption rates of 11  $\text{h}^{-1}$  and 9  $\text{h}^{-1}$  for uranium and thorium, respectively. Nevertheless, these values were obtained under different experimental conditions, with different concentrations of the radioactive material, and using microbial cells, whose specific surface area is much greater than that of the *S. filipendula* biomass. Andres et al. (10) reported thorium biosorption equilibrium for *Mycobacterium smegmatis* at around 120–180 min, which is in line with the present work; however, the experimental conditions were quite different.

### Batch Biosorption Isotherm

The results of the batch biosorption experiments are presented in Fig. 5. These indicate an increasing biosorption of thorium up to a maximum uptake value of 2.59  $\mu\text{mol/g}$  *S. filipendula* biomass.

In a study by Volesky and Yang (5) into the biosorption of uranium by *Sargassum* biomass, they found a maximum uptake capacity of 2.40 mmol/g, which is close to the number of ligand sites present in *Sargassum* biomass (2.25 mmol/g) (11). This value is higher than the uptake achieved in the present work, because the authors were working with a different biomass and radiotracer.

D'Souza et al. (12) studied the biosorption of uranium and thorium by the bacterium *Pseudomonas*. Its maximum uptake capacity was 1.853  $\mu\text{mol Th/g}$  biomass, substantially higher than the results obtained here; however, large-scale biosorption does not seem feasible because most *Pseudomonas* are pathogenic.

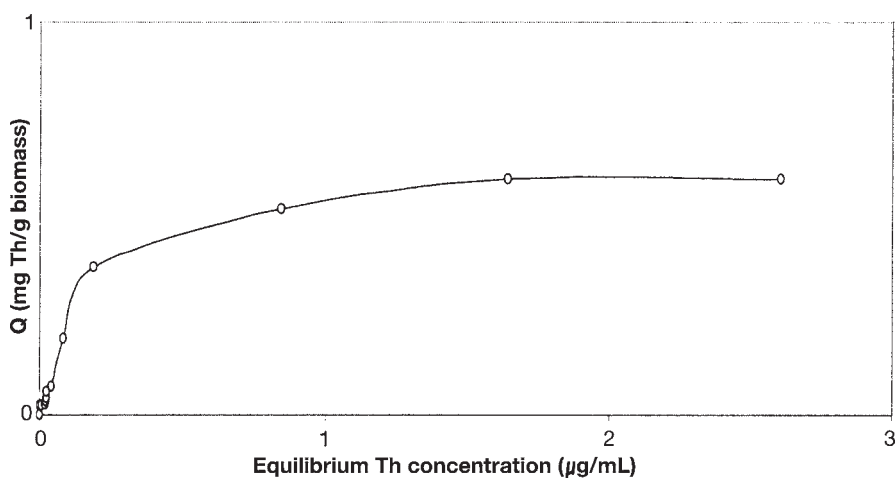


Fig. 5. Thorium biosorption isotherm by dry biomass of *Sargassum filipendula*.

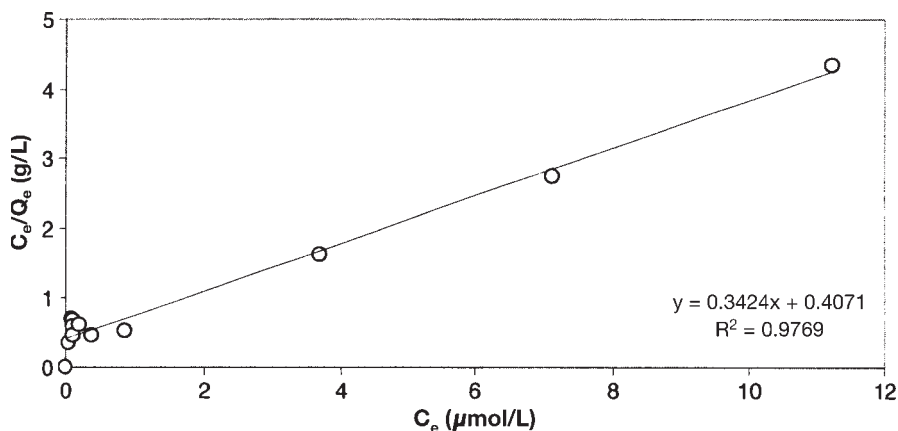


Fig. 6. Thorium biosorption isotherm by *Sargassum filipendula*—Langmuir model.

### Modeling Based on Langmuir and Freundlich Equations

The linearized forms of the Langmuir and Freundlich models, which fit the present experimental results, are presented in Figs. 6 and 7. Although both models presented a good adjustment to the experimental results, the Langmuir model proved more suitable than the Freundlich model, providing a better correlation with the experimental data. This is seen in the high value of the correlation coefficient, as well as the  $Q_0$  value estimated by the model (2.92 µmol/g), which is close to the experimental value obtained (2.59 µmol/g; see Table 2).

The adjustment of experimental data was not so good using the Freundlich model as using the parameters from Langmuir equation, which can be corroborated by the lower value of the correlation coefficient, associated to a low adsorption constant. These values are presented in Table 2.

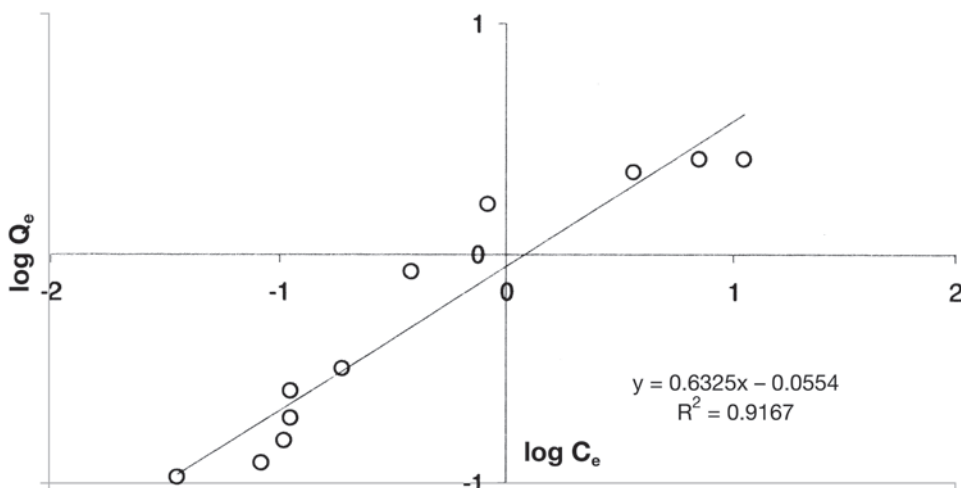


Fig. 7. Thorium biosorption isotherm by *Sargassum filipendula*—Freundlich model.

Table 2  
Constants From the Langmuir and Freundlich Models

$Q_0$ ( $\mu\text{mol/g}$ )	Langmuir constants		Freundlich constants		
	$K_L$ (L/gmol)	$R^2$	$n$	$K_f$	$R^2$
2.92	0.84	0.9769	1.58	0.88	0.9167

Unlike in the present work, Volesky and Yang (5) found the Freundlich model suitable for explaining uranium biosorption by *Sargassum* biomass. Meanwhile, Aslani et al. (4), who studied thermodynamic and kinetic parameters during the biosorption of thorium by a sea sponge, found a good correlation to the Langmuir model and a maximum biosorption capacity of 0.0021  $\mu\text{mol/g}$ .

A good correlation with the Langmuir model suggests the formation of a monolayer between the metal and the biomass. As the monolayer saturates, the uptake of the metal is significantly affected by the transport of the solute.

Valdman and Leite (13) studied cadmium, zinc, and copper biosorption by *Sargassum* biomass in a wide range of concentrations. The authors showed that both the Langmuir and the Freundlich model were suitable to fit the experimental results, with maximum uptake capacities of 140, 182, and 122  $\mu\text{mol/g}$  biomass for cadmium, zinc, and copper, respectively.

Chu and Hashim (14) studied brown, green, and red seaweeds for the biosorption of cadmium. The Langmuir model fitted their results, and they showed that the brown seaweeds presented higher uptake capacities. *Sargassum siliculosum* and *Sargassum baccularia*, both brown seaweeds, accumulated 0.73 and 0.74 mmol/g biomass, respectively.

In a comparative study of several seaweeds, including *Sargassum* sp. (brown), *Padina* sp. (brown), *Ulva* sp. (green), and *Gracillaria* sp. (red), for the uptake of lead, copper, cadmium, zinc, and nickel, Ting and Sheng (15) observed that brown seaweeds presented the best performance. This was attributed to the presence of alginate, which serves as a structural polysaccharide (11).

The results obtained in the present work provide additional information about the biosorption of radioactive thorium, whose performance is comparable to the performance reported in the literature for other heavy metals. In particular, kinetic and sorption parameters were determined, data for which were not encountered in the published literature.

## Conclusions

In previous experiments, the potential use of *S. filipendula* in the biosorption of thorium from several solutions was confirmed, with biosorption rates ranging from 70 to 100%, 70 to 80%, and 20 to 70%, from low, moderate, and high concentrations of thorium in the solution, respectively. The kinetic studies indicated that equilibrium is reached after 3 h of contact between the biomass and thorium solution, which characterizes it as having a slow uptake process. A second-order kinetic model proved suitable to fit the experimental data obtained, given the high correlation coefficient observed and the similarity between the experimental and predicted maximum uptake values. The biosorption isotherm indicated that the maximum thorium uptake capacity would be 2.59  $\mu\text{mol/g}$  biomass. The higher correlation coefficient achieved by the Langmuir adsorption model meant it fitted the experimental results better than the Freundlich model.

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