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# Adsorption of $Ni^{2+}$ from Aqueous Solutions by Pretreated Biomass of Marine Macroalga *Durvillaea potatorum*

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## ABSTRACT

Biosorption of heavy metals can be an effective process for the removal and recovery of heavy metal ions from aqueous solutions. The biomass of marine macroalgae has been reported to have high uptake capacities for a number of heavy metal ions. In this paper the adsorption properties of a pretreated biomass of macroalga *Durvillaea potatorum* for $Ni^{2+}$ were investigated. Adsorption isotherms and kinetics were obtained from batch adsorption experiments. The adsorption capacities were pH dependent and a maximum adsorption capacity was obtained to be 1.13 mmol/g at a pH of about 6. A modified Langmuir model was proposed for the correlation of pH-dependent isotherms. The adsorption process was fast; 90% of adsorption occurred within 25 minutes and equilibrium was reached at around 1 hour. Light metal ions at a concentration of 10 mM did not affect $Ni^{2+}$ adsorption significantly. Fixed-bed breakthrough curves for $Ni^{2+}$ removal were also obtained. This study demonstrated that the pretreated biomass of *D. potatorum* can be used as an effective biosorbent for the treatment of $Ni^{2+}$-containing wastewater streams.

*Key Words.* Biosorption of heavy metals; Marine macroalgae; *Durvillaea potatorum*; Wastewater treatment

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## INTRODUCTION

The removal and recovery of heavy metals from wastewater streams is important in the protection of the environment and human health. Nickel is one of the heavy metals present in raw wastewater streams from industries such as nonferrous metals mineral processing, paint formulation, porcelain enameling, and steam-electric power plants (1, 2). A number of technologies have been used to remove $Ni^{2+}$ from wastewater streams, such as adsorption onto activated carbon (2–5), filtration of precipitate (6), and by crystallization in the form of nickel carbonate (7). However, these technologies are most suitable in situations where the concentrations of the heavy metal ions are high. They are either ineffective or expensive when heavy metals are present in the wastewater at low concentrations or when very low concentrations of heavy metals in treated water are required (8).

Alternative technologies of treating diluted wastewater streams have been studied in recent decades (9, 10). Biosorption is one such technology in which an increasing amount of study is being focused. It utilizes the ability of biological materials to accumulate heavy metals from waste streams by either metabolically mediated or purely physicochemical pathways of uptake (11). Materials that have been investigated for heavy metal uptake include fungi (12, 13), bacteria (14, 15), yeast (16), microalgae (17, 18), macroalgae (19, 20), and others (21–23). Many are available in large quantities either as industrial waste by-products or from natural sources.

Marine macroalga is available in large quantities in many parts of the world. Recently, the use of biomasses of marine macroalgae, *Durvillaea potatorum* (19, 20), *Ecklonia radiata* (19), *Ascophyllum nodosum* (24), and *Sargassam baccularia* (25), for heavy metal removal has been reported. The studies indicate that marine macroalgae can form a good basis for the development of biosorbent materials. Among them, *D. potatorum* is a common brown marine alga found and harvested in the subtidal or intertidal fringe along the rocky coastlines of southern Australia (26). In this paper the biosorption of $Ni^{2+}$ ions from aqueous solutions by using a pretreated biomass of *D. potatorum* is studied in laboratory-scale experiments. The biosorbent was pretreated by a two-stage process of thermal and chemical modification of the native biomass (19, 20). This study showed that the biomass can accumulate a high amount of $Ni^{2+}$ from aqueous solutions and that it might be a suitable biosorbent for practical metal recovery applications. However, regeneration of the biosorbent for reuse and the investigation of industrial-scale applications are not included in the scope of this paper.

## MATERIALS AND METHODS

Sun-dried *D. potatorum* samples in ground form were supplied by Kelp Industries Pty. Ltd. Tasmania, Australia. Pretreatment of the biomass was carried out as follows: A sample of 20 g of biomass was treated with 0.2 M $CaCl_2$ solution (400 mL) for 24 hours under slow stirring. The solution pH was kept constant at pH 5.0 by using 0.1 M $HNO_3$ or 0.1 M NaOH solution if deviations were observed. The calcium-treated biomass was washed several times with deionized water to remove excess calcium. The biomass was then heated in an oven at 60°C for 24 hours and then sieved for a particle size of 300–600 μm. This pretreatment improved the chemical and mechanical stability of the biosorbents by reducing the amount of total soluble organic carbon in water extraction, the swelling volumes, and the attrition loss (27).

All experiments were conducted at room temperature (25 ± 2°C). For isotherm determination, a series of 125 mL plastic vials were prepared containing nickel nitrate solutions (100 mL) of known concentrations, which varied from 0.5 to 4.5 mM. Weighed amounts [200 mg (dry)] of biomass were added to each vial, and the mixtures were agitated on a rotary shaker for 24 hours. The solution pH was adjusted to the required value by using 0.1 M $HNO_3$ or 0.1 M NaOH hourly in the first 4 hours. The pH of the solution was measured and adjusted if necessary again after 24 hours (no further changes in pH were observed). The biomass was removed by filtration through a 0.45-μm membrane filter (which does not adsorb the heavy metal ions), and the filtrates were analyzed for $Ni^{2+}$ by atomic absorption spectrometry.

Kinetics experiments were conducted in continuously stirred beakers (200 rpm) containing 500 mL of solution and 1 g biomass. Samples of 1 mL each were drawn from the mixture at predetermined time intervals for analysis. The pH of the solution was monitored continuously with a pH electrode and adjusted with 0.1 M $HNO_3$ or 0.1 M NaOH solution if deviations were observed.

Fixed-bed experiments were performed in a glass column of 1 cm diameter, packed with 1 g biomass (bed volume 6.6 mL). Solutions of $Ni^{2+}$ were pumped through the column at a flow rate of 1.5 mL/min using a peristaltic pump (Gilson Miniplus-2). Effluent samples were collected for every bed volume using a fraction collector (Gilson FC-203) and were analyzed for $Ni^{2+}$ concentration by using atomic absorption spectrometry.

## ADSORPTION ISOTHERM MODELS

The standard Langmuir adsorption model was used to correlate the isotherm data obtained at constant solution pH values,

$$q = \frac{bQ_{\max}c}{1 + bc} \tag{1}$$

where $q$ is the amount of $Ni^{2+}$ adsorbed at equilibrium (mmol/g), $c$ is the $Ni^{2+}$ ion concentration in solution (mM) at equilibrium, $Q_{\max}$ is the maxi-

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mum adsorption capacity, and $b$ is an affinity constant. For determining the equilibrium parameters, Eq. (1) may be written into a linearized form as follows:

$$\frac{c}{q} = \frac{1}{Q_{\max} b} + \frac{c}{Q_{\max}} \tag{2}$$

The parameters were then obtained by using a least square linear regression analysis.

In Equation (1) the parameters $b$ and $Q_{\max}$ are solution pH dependent. Thus the application of Eq. (1) is limited to isotherms obtained at constant pH values. However, heavy metal biosorption is strongly affected by the solution pH (12, 16, 23, 25). In addition, the biosorption process normally affects solution pH, and variations were observed as the heavy metal ions were adsorbed. In order to take this phenomenon into account, a modified Langmuir model was proposed and used to correlate isotherm data obtained at various solution pH values. In this model it is assumed that the functional groups responsible for heavy metal interaction are weakly acid groups. The ionic forms of the functional groups are therefore affected by the association–dissociation equilibrium. It is further assumed that the weak acid equilibria can be represented by the following apparent single equilibrium:

$$\mathrm{GH} \xleftrightarrow{K_a} \mathrm{G}^- + \mathrm{H}^+ \tag{3}$$

where GH and $\mathrm{G}^-$ represent the protonated and deprotonated forms of the functional groups. Finally, it is assumed that the maximum adsorption capacities for GH and $\mathrm{G}^-$ are $Q_{\max1}$ and $Q_{\max2}$, respectively.

From the equilibrium in Eq. (3), the overall maximum adsorption capacity for the biomass is the weighted average of the two ionic forms of the functional groups,

$$Q_{\max} = \frac{Q_{\max1} + Q_{\max2}\, 10^{\mathrm{pH}-\mathrm{p}K_a}}{1 + 10^{\mathrm{pH}-\mathrm{p}K_a}} \tag{4}$$

where $\mathrm{p}K_a$ is the equilibrium constant for Eq. (3).

Equation (4) gives the pH-dependant $Q_{\max}$ values which can be substituted into Eq. (1) for the correlation of pH dependent isotherm data.

By combining Eqs. (4) and (1) and rearrangement, the following linearized form can be obtained:

$$C = \frac{-1}{b} + \left(\frac{C}{q}\right)\left(\frac{1}{1 + 10^{\mathrm{pH}-\mathrm{p}K_a}}\right) Q_{\max1} + \left(\frac{C}{q}\right)\left(\frac{10^{\mathrm{pH}-\mathrm{p}K_a}}{1 + 10^{\mathrm{pH}-\mathrm{p}K_a}}\right) Q_{\max2} \tag{5}$$

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In this study the values of parameters $b$, $Q_{\max1}$, and $Q_{\max2}$ were obtained from a multi-linear regression analysis by using Eq. (5) at a fixed value of p$K_a$, and the value of p$K_a$ was determined from trial and error.

## RESULTS AND DISCUSSION

### Effect of Solution pH on Heavy Metal Adsorption

Earlier studies indicated that solution pH strongly affects biosorption of heavy metal ions (12, 16, 23, 25). This effect of pH on $Ni^{2+}$ adsorption by a pretreated biomass of *D. potatorum* was therefore studied first. The amount of $Ni^{2+}$ adsorption as a function of solution pH is shown in Fig. 1. At a pH of less than 2, the amount of $Ni^{2+}$ uptake is small. As pH increases, the amount of uptake increases and the sharpest increase is observed between pH 3 and 5. At around pH 6 a plateau is reached. These results show the strong pH dependence of $Ni^{2+}$ biosorption. Experiments were not conducted beyond pH 6 to avoid possible heavy metal precipitation. Preliminary experiments for possible precipitation of $Ni^{2+}$ near pH 6 in the absence of biosorbents were also conducted. No precipitation was observed.

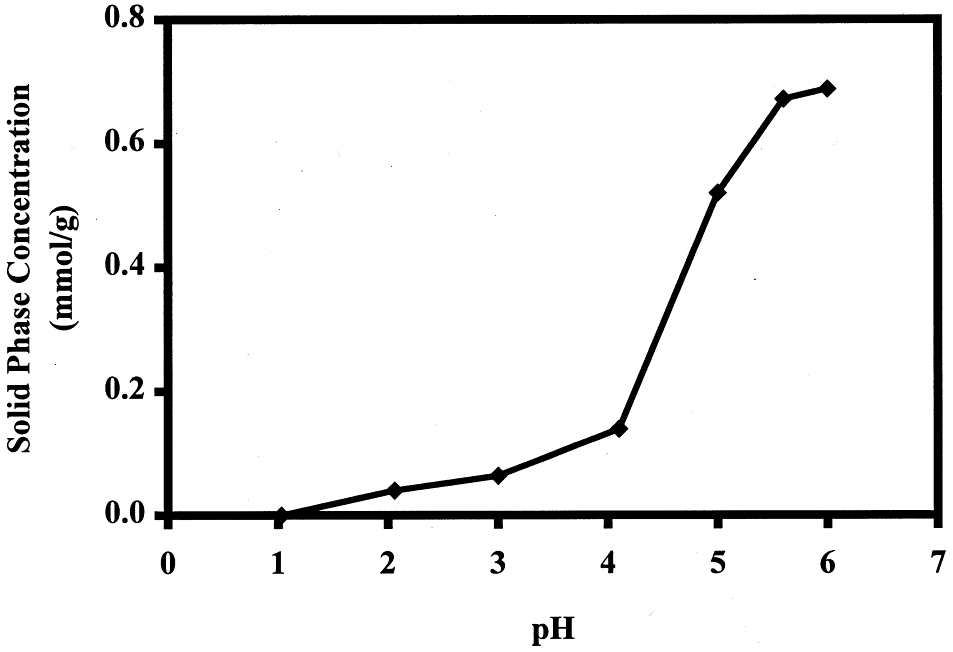

FIG. 1 Amount of $Ni^{2+}$ adsorption by pretreated biomass of *D. potatorum* at various pH (adsorbent dose, 2 g/L; initial $Ni^{2+}$ concentration, 2 mM).

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## Adsorption Isotherms

Adsorption isotherms were obtained under constant solution pH values and are shown in Fig. 2. The isotherms are rather steep at lower equilibrium concentrations, implying high adsorption affinity and thus the suitability of biosorbents to treat dilute heavy metal solutions. The adsorption isotherm plots have the typical shape of L-2 type isotherms, indicating a reduction in the number of active sites on the adsorbents at a high residual heavy metal concentration in the solution phase.

The isotherms in Fig. 2 were described by the standard Langmuir equation (Eq. 1). From the linearized Langmuir plots (Eq. 2), the isotherm constants were derived by performing a linear regression analysis on each set of isotherm data and are presented in Table 1. The isotherms were also correlated by the modified Langmuir equation (Eq. 4) and are plotted in Fig. 3. From the linearized modified Langmuir plots with a best-fit value for $pKa = 5.05$ and a multiple variable regression analysis using SPSS, the isotherm constants were obtained and are also presented in Table 1.

Table 1 shows the adsorption capacity $Q_{\max}$ increases with an increase in solution pH, supporting the effect of pH on $Ni^{2+}$ adsorption. It is noted that the value of $Q_{\max1}$ is close to but less than that of $Q_{\max}$ at pH 3, and the value

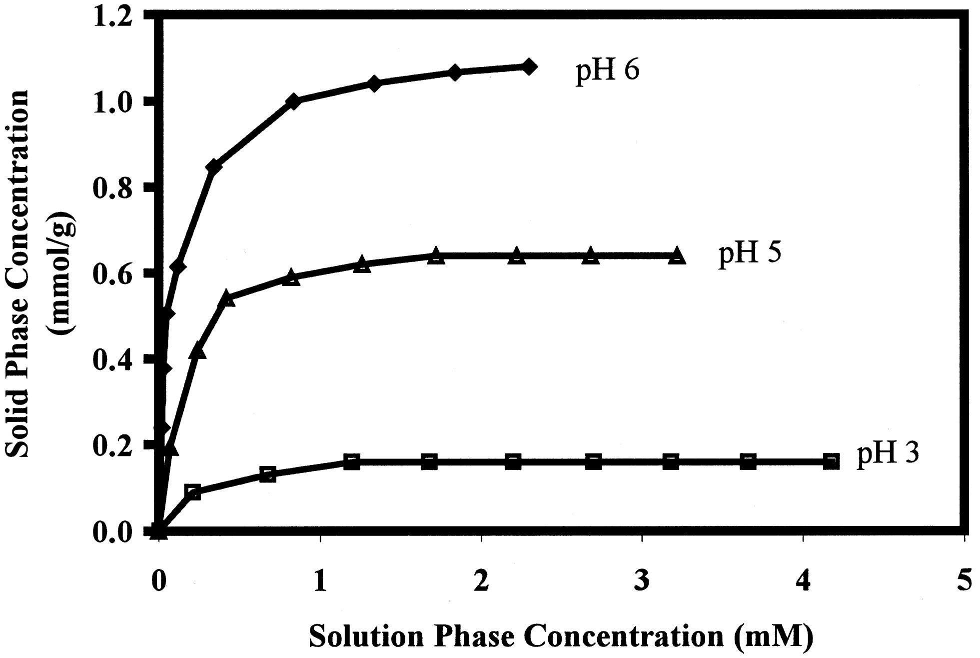

FIG. 2 Adsorption isotherms of $Ni^{2+}$ onto pretreated biomass of *D. potatorum* at various pH with the Langmuir model (solid lines).

TABLE 1
Langmuir Isotherm Constants for $Ni^{2+}$ Adsorption

| Equation | pH | $Q_{max}$ (mmol/g) | $b$ (1/mM) | $r^2$ |
|---|---|---|---|---|
| 1 | 3 | 0.17 | 6.23 | 0.999 |
| 1 | 5 | 0.68 | 7.98 | 0.999 |
| 1 | 6 | 1.13 | 8.05 | 0.999 |
| 1 and 4 | All pH | $Q_{max1} = 0.15$<br>$Q_{max2} = 1.23$ | 8.96 | 0.998 |

of $Q_{max2}$ is close to $Q_{max}$ at pH 6. This is consistent with the assumption the protonated forms of the functional groups have low metal binding abilities, and metal binding is related to the electrostatic interactions between the metal ions and the functional groups.

A comparison of the maximum $Ni^{2+}$ adsorption capacity ($Q_{max}$) of *D. potatorum* with those of other adsorbents as reported in the literature is given in

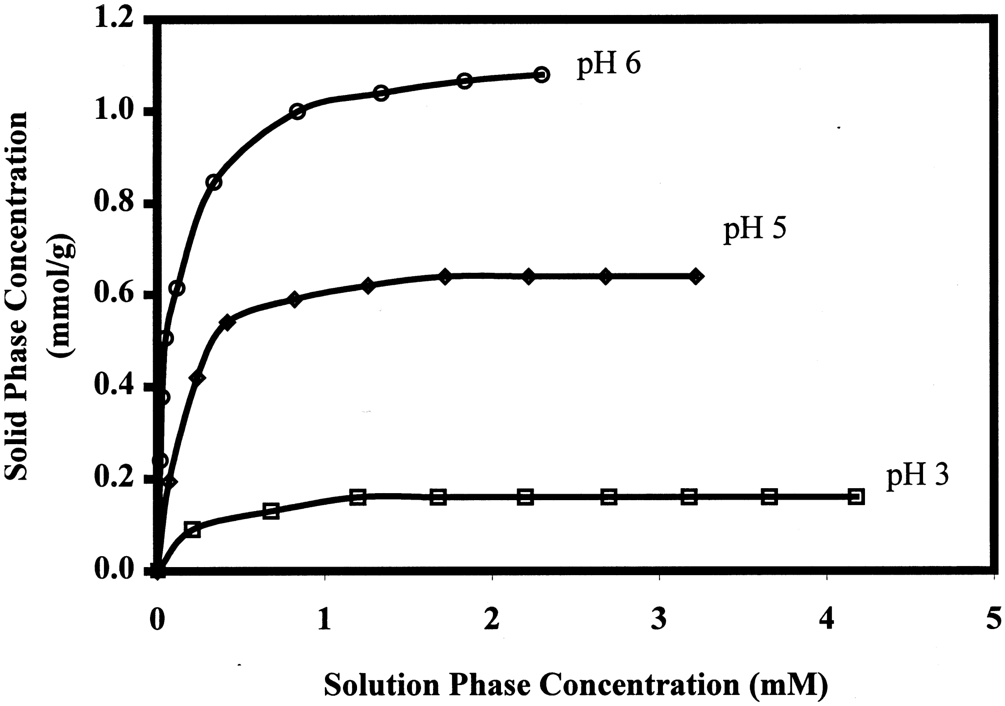

FIG. 3 Adsorption isotherms of $Ni^{2+}$ onto pretreated biomass of *D. potatorum* at various pH with the modified Langmuir model (solid lines).

TABLE 2
Uptake Capacities ($Q_{max}$, mmol/g) for $Ni^{2+}$ of Various Adsorbents

| Adsorbent | $Q_{max}$ (mmol/g) |
|---|---|
| Rice hulls (28) | 0.098 |
| Sphagnum moss peat (29) | 0.405 |
| Sphagnum moss peat (30) | 0.161 |
| *R. nigricans* (fungal organisms) (31) | 0.070–0.0704 |
| *A. orchidis* (fungal organisms) (32) | 0.070–0.0704 |
| Anaerobically digested sludge (33) | 0.1348 |
| China clay (34) | 0.061 |
| Amorphous iron hydroxide (35) | 0.119 |
| Oil-palm fiber (36) | 0.009 |
| Sludge solids (37) | 0.718 |
| *D. potatorum* (this study) | 1.13 |

Table 2. The adsorption capacity of *D. potatorum* (1.13 mmol/g) is considerably higher than many of these adsorbents.

## Adsorption Kinetics

Figure 4 presents a typical set of results from kinetic experiments of $Ni^{2+}$ adsorption onto the biomass of *D. potatorum* at different initial concentrations. It was observed that 90% of the total soluble $Ni^{2+}$ was removed from solutions within 25 minutes of contact time. This initial rapid metal uptake has significant practical importance as it will facilitate shorter adsorption columns, thus ensuring efficiency and economy. Afterwards, there were slower rates of uptake to about 1 hour, and no further significant adsorption was observed beyond this period. This was the same for all the initial metal concentrations for the *D. potatorum* biomass studied.

## Effects of Light Metal Ions

The effect of the presence of $Ca^{2+}$, $Mg^{2+}$, $Na^{+}$, and $K^{+}$ ions (one at a time) in the solution on the $Ni^{2+}$ uptake capacity of the biosorbents was investigated. These light metal ions were considered as quite likely to be present in solutions originating from an industry source. A concentration of 10 mM of the light metal ions was used to study their effect. The results are compared in Fig. 5. It is seen from the figure that the effect of $Na^{+}$ on $Ni^{2+}$ uptake was negligible at a concentration of 10 mM, and $K^{+}$, $Mg^{2+}$, and $Ca^{2+}$ reduced the removal efficiency by 2, 5, and 8%, respectively. This could be a significant ad-

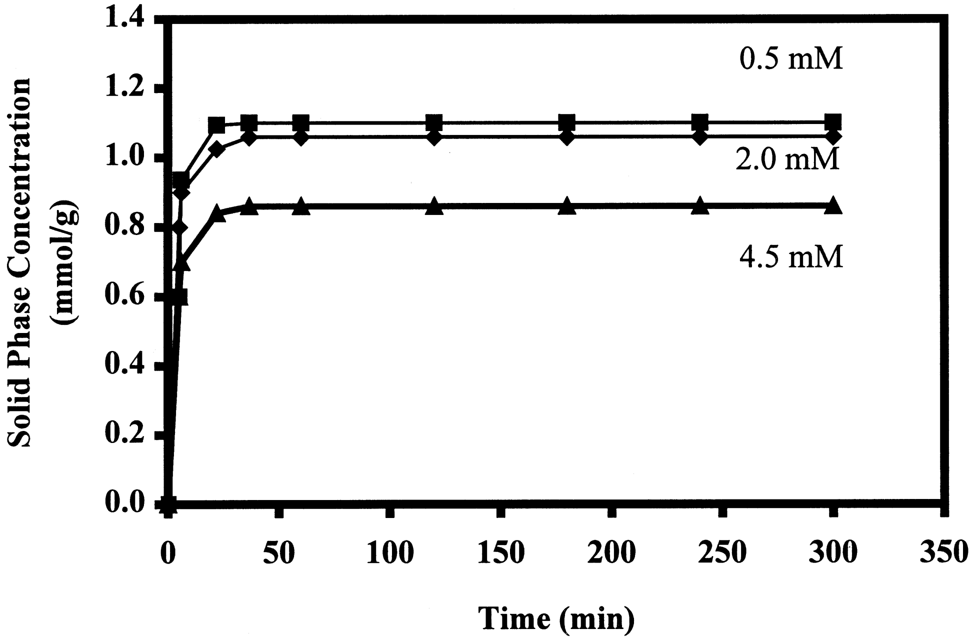

FIG. 4 Adsorption kinetics of $Ni^{2+}$ onto pretreated biomass of *D. potatorum* with various initial concentrations (adsorbent dose, 2 g/L; agitation speed, 200 rpm; solution pH kept at 6).

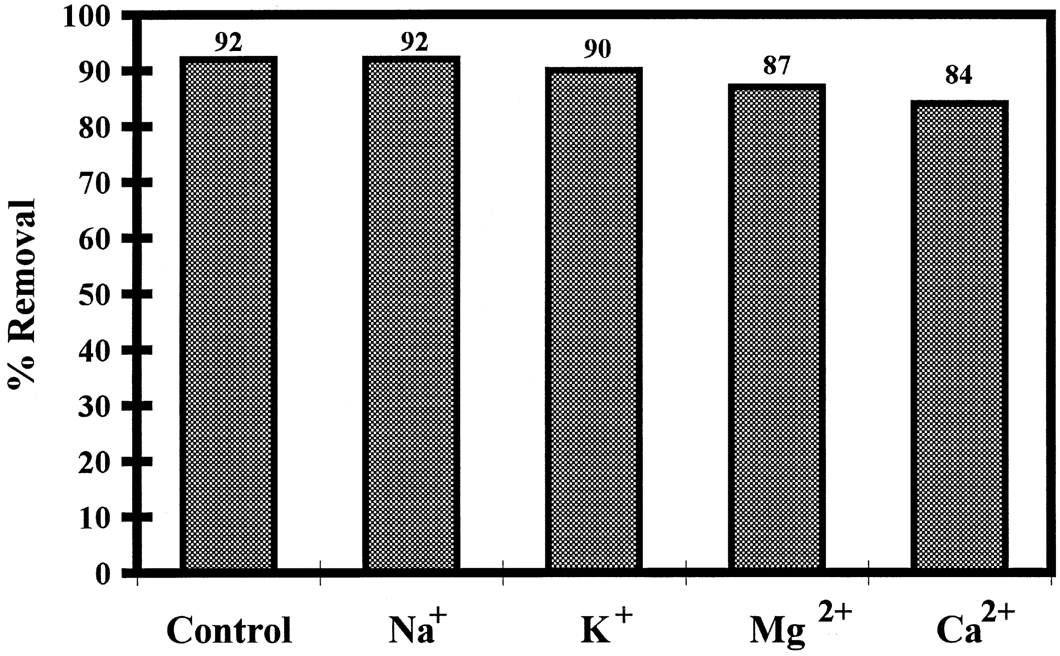

FIG. 5 Removal efficiencies of $Ni^{2+}$ by *D. potatorum* in the presence of various light metal ions ($Ni^{2+}$ concentration, 2 mM; adsorbent dose, 2 g/L; solution pH kept at 6).

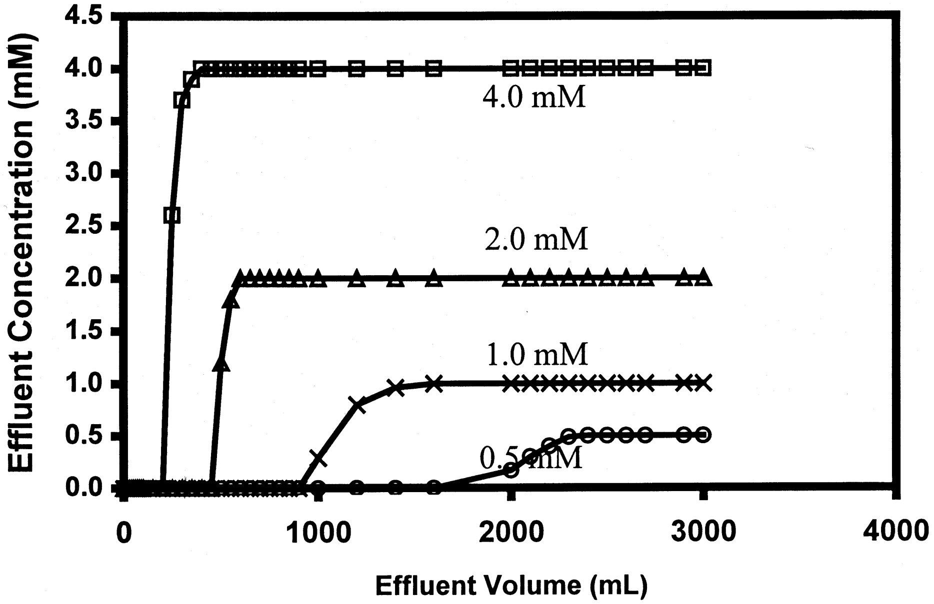

FIG. 6 Fixed-bed breakthrough curves for $Ni^{2+}$ removal by *D. potatorum* with various feed concentrations (adsorbent dose, 2 g; column diameter, 1 cm; bed volume, 6.6 mL; flow rate, 1.5 mL/min; empty bed contact time, 4.4 minutes).

vantage of algal adsorbents over the commercially available ion-exchange resins because the binding of $Ca^{2+}$ and $Mg^{2+}$ (which are normally present in high concentrations in industrial wastewater) to these resins often reduces their efficiencies.

## Fixed-Bed Breakthrough Curves

Fixed-bed breakthrough curves at various feed concentrations of $Ni^{2+}$ were obtained to illustrate the suitability of column operations (Fig. 6). The breakthrough curves followed the typical S-shaped curve for column operation with favorable adsorption isotherms. An adsorption column containing 1 g (dry weight) of the biomass could purify 2.0 L of 0.5 mM nickel solution before breakthrough, and it purified 1.0, 0.5, and 0.25 L of the solutions when the feed concentrations were 1.0, 2.0, and 4.0 mM, respectively. The total uptake capacities of the fixed bed for the different initial concentrations were calculated by integrating the breakthrough curves between the breakthrough and saturation points. The adsorption capacities of the column for various initial concentrations ranged between 1.04 and 1.13 mmol/g, which agrees well with the maximum value of 1.13 obtained from batch experiments.

## CONCLUSIONS

The study indicated that the pretreated biomass of *D. potatorum* could be used as an efficient biosorbent material for the treatment of Ni$^{2+}$-ion-bearing wastewater streams. The adsorption capacities were solution pH dependent, and a maximum adsorption capacity of 1.13 mmol/g was obtained at a solution pH of about 6. The adsorption capacity for Ni$^{2+}$ of *D. potatorum* was found to be much higher than those of many other adsorbent materials reported in heavy metal recovery and removal applications. The kinetics of adsorption by this biomass is rapid with 90% of total adsorption occurring within 25 minutes. The presence of 10 mM Ca$^{2+}$, Mg$^{2+}$, Na$^{+}$, and K$^{+}$ did not significantly interfere with the binding of Ni$^{2+}$ ions. The biomass can be used in fixed-bed operations.

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