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# Sorption of Heavy Metal Ions by the Nonliving Biomass of Freshwater Macrophytes

IVO ANDRÉ H. SCHNEIDER*,† AND JORGE RUBIO‡

*FEAR, Universidade de Passo Fundo, CEP 99001-970, Passo Fundo, RS, Brazil, and DEMIN, Universidade Federal do Rio Grande do Sul, CEP 90035-190, Porto Alegre, RS, Brazil*

The removal of heavy metal ions by the nonliving biomass of aquatic macrophytes was investigated. The work involved studies of physical and biochemical properties of the materials, batch sorption experiments carried out in agitation flasks, and continuous runs in a packed bed column at laboratory scale. Results showed that the dried biomass of *Potamogeton lucens*, *Salvinia herzogii,* and *Eichhornia crassipes* were excellent biosorbents for Cr-(III), Ni(II), Cu(II), Zn(II), Cd(II), and Pb(II). The sorption mechanism by these biomaterials was found to proceed mainly by ion exchange reactions between the metal ions and the cationic weak exchanger groups present on the plant surface. Sorption followed the Langmuir isotherm, and maximum metal uptakes values (independently of the metal ion species) were attained at about 1.5 mequiv $g^{-1}$ for *P. lucens*, 0.9 mequiv $g^{-1}$ for *S. herzogii,* and 0.7 mequiv $g^{-1}$ for *E. crassipes*. Advantages and disadvantages found in the use of these natural adsorbents for heavy metals ions present in industrial wastewaters are envisaged.

## Introduction

During the past two decades, there has been interest in the use of aquatic plants in treating polluted effluents. Much of the attention has been addressed to *Eichhornia crassipes* (water hyacinth), a free floating weed of worldwide distribution (*1*−*11*). The rapid, often excessive, growth of this plant allows a biomass production that far exceeds the yield of most productive agricultural crops. Other species studied have been *Alternanthera philoxeroides* (alligator weed) (*2*, *3*), *Pistia stratiotes* (water lettuce) (*11*), and *Potamogeton crispus (12*).

Early studies concerning the use of *E. crassipes* and other aquatic plants in pond systems to remove metal ions and other pollutants from wastewaters were conducted by the National Space Technology Laboratories (NASA/NSTL) in United States in the 1970s (*1*−*5*). It was demonstrated at laboratory scale and at a wastewater treatment station that this weed efficiently removes dilute concentrations of heavy metals, including Co, Ni, Sr, Ag, Cd, Hg, and Pb. In the pond system used, metal-saturated water hyacinths were periodically harvested and disposed off in adjacent pits, which were designed to prevent heavy metal leaching from the decayed plant material.

* Corresponding author e-mail: ivoandre@upf.tche.br; phone: 55 54 3168201; fax: 55 54 3168211.

† Universidade de Passo Fundo.

‡ Universidade Federal do Rio Grande do Sul.

Later, several other studies dealt with the heavy metal removal capacity of living macrophytes. Most of these studies were carried out in plastic or glass containers in greenhouses, and the main subjects of investigation were in the field of solution chemistry and sorption kinetics (*6*−*10*). The plants placed in these static systems were able to remove the metals in a few hours of exposure. However, it has been demonstrated that the ions produce phytotoxic effects on plants resulting in inhibition of chlorophyll synthesis, decrease in biomass production, and finally plant necrosis (*11*−*13*). For these reasons and because of disposal problems of contaminated plants, the direct application of water hyacinths and other aquatic plants in wastewater decontamination is being neglected.

Conversely, the metal sorption capacity of the dried biomass of aquatic plants has been recently recognized (*14*). The main advantages in using the dead biomass instead of living systems appears to be the following:

(a) problems of metal toxicity on plant metabolism, plant deterioration, odor liberation, and insects proliferation are avoided;

(b) the dried biomass presents advantages for conservation, transport, and handling and as such becomes ready for usage in wastewater units as a simple sorbent material;

(c) it is possible to recover the sorbed heavy metals by elution techniques using sorption/desorption cycles.

This paper investigates the sorption characteristics (mechanism and applications) of heavy metal ions by selected and low-cost biomasses prepared from harvested freshwater macrophytes. The main features involving the sorption of the ions onto plant tissues and the advantages for use of these materials in water treatment are discussed.

## Materials and Methods

**Metal Solutions.** Salts used in the preparation of the synthetic metal bearing solutions were of analytical grade: $Cr_2(SO_4)_3$, $NiSO_4 \cdot 6H_2O$, $CuSO_4 \cdot 5H_2O$, $ZnSO_4 \cdot 7H_2O$, $CdSO_4 \cdot 8/3H_2O$, and $Pb(NO_3)_2$. Medium pH values were adjusted using reagent grade NaOH and $HNO_3$. Deionized water was used in all experiments.

Effluents containing residual amounts of heavy metals were obtained from two different industries: a copper sulfate production plant and a typical electroplating industry.

**Biosorbents.** Aquatic macrophytes *Cabomba* sp., *Ceratophyllum demersum*, *Myriophyllum brasiliensis*, *Eichhornia crassipes*, *Potamogeton lucens*, and *Salvinia herzogii* were obtained from wild specimens growing in Rio Grande do Sul State, Brazil. The plant tissues were washed in deionized water, dried at 60 °C, and ground in a bladed mixer to less than 0.59 or 4.0 mm.

**Biomass Characterization.** Bulk density was measured according to ASTM Standards D5057-90, and apparent density was measured by picnometry using ethylic alcohol 96°GL. Water retention, in grams of water per grams of biosorbent, was determined by the difference in the wet and dry weight forms of the material. The wet weight was obtained by soaking the material in water for a few minutes and then removing the water excess by aspiration in a filter crucible.

The surface area was measured using the dye adsorption method (*15*). The surface area was calculated from the saturation adsorption assuming a cross sectional area of 1.6 $nm^2$ for the rhodamine B molecule.

The biochemical composition of the biosorbents was characterized in terms of proteins, carbohydrates, lipids, and ash according to AOAC (*16*). Determination of the amount of protein from the Kjeldahl nitrogen used a multiplication

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value for the nitrogen−protein ratio of 6.25. The carbohydrate content was calculated from the difference between the total weight of solids and the weight of protein, lipids, and ash.

A technique described by Simon (*17*) was used to measure (qualitatively) the ion exchange properties of the sorbent materials. Carboxyl groups were determined with $Ba(CH_3COO)_2$ (*18*), and the total acidity was determined by the $Ba(OH)_2$ method (*19*). The amount of phenolic hydroxyl groups was determined by the difference between the total acidity and the carboxyl content.

**Batch Sorption Studies.** The metal ions uptake from aqueous solutions by the biosorbents was measured by placing 100 mL of the ion solution into contact with a preweight of biosorbent under stirring. Preliminary experiments of adsorption kinetics indicated that a period of 30 min was sufficient to attain equilibrium. Separation of the suspended biosorbents was performed by filtration in polypropylene disks to avoid metal uptake by the filters. The concentration of metal ions in solution and the medium pH were determined both before and after biosorption by flame atomic absorption spectroscopy and with a pH meter, respectively. Each experiment was repeated in duplicate runs, both producing satisfactory results.

Sorption isotherms were measured at 25 °C by varying the initial metal ion concentration and keeping the biosorbent mass constant. Isotherms were analyzed by the Langmuir adsorption model, which describes the sorption reaction as

$$m = \frac{MbC}{(1 + bC)} \tag{1}$$

or by its linearized form

$$\frac{C}{m} = \frac{1}{bM} + \frac{C}{M} \tag{2}$$

where $m$ is the mass of solute sorbed per unit weight of sorbent, $M$ is the mass of solute sorbed per unit weight of sorbent at saturation, $b$ is the constant accounting for the energy of interaction, and $C$ is the measured concentration in solution at equilibrium.

**Packed Bed Column Studies.** Sorption/desorption experiments were performed in a cylindrical column of 2.5 cm diameter with 4 g of adsorbent having particle size less than 4.0 mm (bed vol of about 0.04 L). Each sorption cycle was carried out with 24 L of a copper solution of 12 mg $L^{-1}$. The flow rate used was about 60 mL $min^{-1}$ or 1.5 bed vol/min. After saturation, the biomass was regenerated with a 0.5% HCl solution and neutralized with an aqueous solution at pH 8.8 ± 0.2.

The metal uptake in a sorption cycle in the packed bed column was calculated by the following expression:

$$M = \frac{VC_oF_a}{m} \tag{3}$$

where $M$ is the heavy metal uptake at saturation, $V$ is the total volume of water treated to the breakthrough, $C$ is the metal concentration in the liquid at any volume, $C_o$ is the initial metal concentration in the liquid, $F_a$ is the ratio between the dotted and the total area described by the breakthrough, and $m$ is the mass of biosorbent. The same experimental conditions were used to treat the industrial effluents at laboratory scale.

## Results

Sorption results of copper by the biomass of different macrophytes are shown in Table 1. Best removal values were obtained with *P. lucens*, followed by *S. herzogii*, *E. crassipes*, and *M. brasiliensis*. The results obtained by *Cabomba* sp. and *C. demersum* were not promising. Very similar sorption data were obtained for the metals nickel and zinc. Thus, the biomass of plants *P. lucens*, *S. herzogii*, and *E. crassipes* were selected for more detailed studies.

**TABLE 1. Sorption of Copper by Selected Freshwater Macrophytes[a]**

| aquatic macrophyte | initial concn (mg $L^{-1}$) | final concn (mg $L^{-1}$) | removal (%) |
|---|---|---|---|
| *Potamogeton lucens* | 6.3 | 0.3 | 95 |
| *Salvinia herzogii* | 6.3 | 0.4 | 94 |
| *Eichhornia crassipes* | 6.3 | 1.3 | 79 |
| *Myriophyllum brasiliensis* | 6.3 | 1.4 | 78 |
| *Cabomba* sp. | 6.3 | 3.4 | 46 |
| *Ceratophyllum demersum* | 6.3 | 3.9 | 38 |

[a] 2 g of biosorbent $L^{-1}$, initial pH 5.5 ± 0.2, 30-min reaction with agitation.

Some of the physical and biochemical properties of the biomass of *P. lucens*, *S. herzogii*, and *E. crassipes* are summarized in Table 2. It was observed that the materials, when suspended in water, absorb (swells) water "restoring" partially their initial situation (aqua). *P. lucens* particles settle very rapidly in water, and *S. herzogii* and *E. crassipes* split into two portions: one that settles (mainly roots) and another that floats (mainly leaves). All biosorbents present high surface areas yielding 415 $m^2$ $g^{-1}$ for *P. lucens*, 270 $m^2$ $g^{-1}$ for *S. herzogii*, and 250 for $m^2$ $g^{-1}$ for *E. crassipes*.

The biochemical analysis showed that *P. lucens* has the higher concentration of proteins, while the higher concentration of lipids and carbohydrates were found in *S. herzogii*. Results of quantitative measurements of the concentration of carboxyl and hydroxyl groups are also depicted in the same table. These groups are considered responsible by the weak cation exchange behavior of the biomaterials and probably by the metal sorption capacity. Conversely, anionic exchange properties were not detected in any of the sorbents.

Figure 1 presents sorption isotherms of chromium(III), nickel, copper, zinc, cadmium, and lead by *P. lucens*. These isotherms fit the Langmuir adsorption model as exemplified for copper in Figure 2. The correlation coefficient and the constants of the Langmuir model obtained for all metals and plants studied in this work are summarized in Table 3. The mass of heavy metal sorbed per unit weight of biosorbent ($M$) at saturation is expressed in milligrams per gram, millimole per gram, and milliequivalent per gram. According to the isotherms constants calculated for the $Cu^{2+}$ uptake by the biomasses at pH 5.5, the comparative sorption capacities at high copper concentration solutions were found to be *P. lucens* > *E. crassipes* > *S. herzogii*. Conversely, at low copper concentration solutions the sequence was *P. lucens* > *S. herzogii* > *E. crassipes*. These differences are explained by the different isotherms slopes of the three biosorbents.

The chemical analysis of the solution after the sorption reaction was performed. This was carried out with the biomass in the non-hydrogen form, and the solution contained 100 mg of copper $L^{-1}$ in deionized water (Table 4). After equilibrium, high copper removal from the system occurred, and several other cations were released to solution. The ionic balance showed that *P. lucens* sorbed 0.633 mequiv of $Cu^{2+}$ $g^{-1}$ and released 0.621 mequiv $g^{-1}$ in the form of $Na^+$, $K^+$, $Mg^{2+}$, $Ca^{2+}$, $Fe^{3+}$, and $Mn^{2+}$. Still, other elements present in minor concentration in the biomass, not considered in the balance, were also exchanged by copper.

Figure 3 shows the effect of equilibrium pH on sorption of copper onto *P. lucens*, *S. herzogii*, and *E. crassipes* biomass. Maximum removal was attained between pH 5.5 and pH 6.6 when copper is mainly in the form of the ionic species $Cu^{2+}$ as shown in the speciation diagram for a 6.3 mg of copper

**TABLE 2. Physical and Biochemical Properties of the Biosorbents**

| property | *P. lucens* | *S. herzogii* | *E. crassipes* |
|---|---|---|---|
| particle size | < 0.59 mm | < 0.59 mm | < 0.59 mm |
| bulk density | 0.15 g $cm^{-3}$ | 0.13 g $cm^{-3}$ | 0.13 g $cm^{-3}$ |
| density | 1.2 g $cm^{-3}$ | 1.1 g $cm^{-3}$ | 1.1 g $cm^{-3}$ |
| water retention | 3.1 g $g^{-1}$ | 4.2 g $g^{-1}$ | 3.2 g $g^{-1}$ |
| specific surface area | 415 $m^2$ $g^{-1}$ | 270 $m^2$ $g^{-1}$ | 250 $m^2$ $g^{-1}$ |
| proteins | 21.7% | 11.5% | 10.0% |
| carbohydrates | 66.0% | 77.2% | 69.0% |
| lipids | 0.9% | 1.1% | 0.7% |
| ash | 11.4% | 10.2% | 20.3% |
| ion exchange behavior | cationic weak | cationic weak | cationic weak |
| carboxyl groups | 1.5 mequiv $g^{-1}$ | 0.9 mequiv $g^{-1}$ | 0.7 mequiv $g^{-1}$ |
| phenolic hydroxyl | 1.3 mequiv $g^{-1}$ | 2.2 mequiv $g^{-1}$ | 0.9 mequiv $g^{-1}$ |

**TABLE 3. Langmuir Adsorption Model Constants Obtained from Isotherms**

| biosorbent, metal | mL | *b* (L $mg^{-1}$) | *M* (mg $g^{-1}$) | *M* (mmol $g^{-1}$) | *M* (mequiv $g^{-1}$) |
|---|---|---|---|---|---|
| *P. lucens,* Pb | 0.854 | 0.25 | 141.0 | 0.68 | 1.36 |
| *P. lucens,* Cd | 0.998 | 0.40 | 61.4 | 0.55 | 1.10 |
| *P. lucens,* Zn | 0.841 | 0.19 | 32.4 | 0.50 | 1.00 |
| *P. lucens,* Cu | 0.998 | 0.30 | 40.8 | 0.64 | 1.28 |
| *P. lucens,* Ni | 0.961 | 0.22 | 22.9 | 0.39 | 0.78 |
| *P. lucens,* Cr(III) | 0.718 | 1.65 | 22.4 | 0.43 | 1.29 |
| *S. herzogii,* Zn | 0.972 | 0.28 | 18.1 | 0.27 | 0.55 |
| *S. herzogii*, Cu | 0.997 | 0.44 | 19.7 | 0.31 | 0.62 |
| *S. herzogii,* Ni | 0.994 | 0.37 | 14.4 | 0.24 | 0.49 |
| *E. crassipes*, Zn | 0.998 | 0.22 | 19.2 | 0.29 | 0.59 |
| *E. crassipes*, Cu | 0.998 | 0.23 | 23.1 | 0.36 | 0.73 |
| *E. crassipes,* Ni | 0.995 | 0.23 | 11.6 | 0.20 | 0.40 |

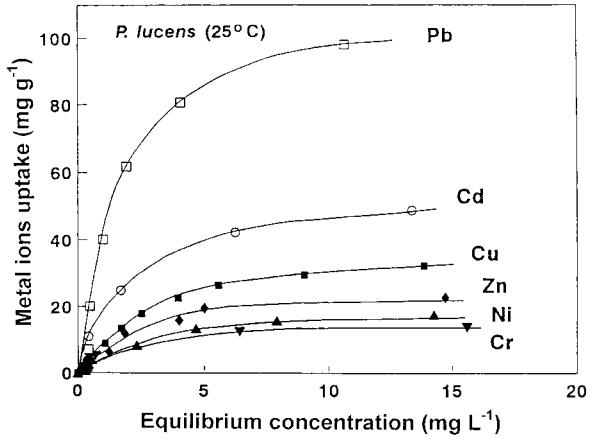


**FIGURE 1. Sorption isotherms of Cr(III), Ni, Cu, Zn, Cd, and Pb on *P. lucens* biomass (initial pH 5.5 ± 0.2).**

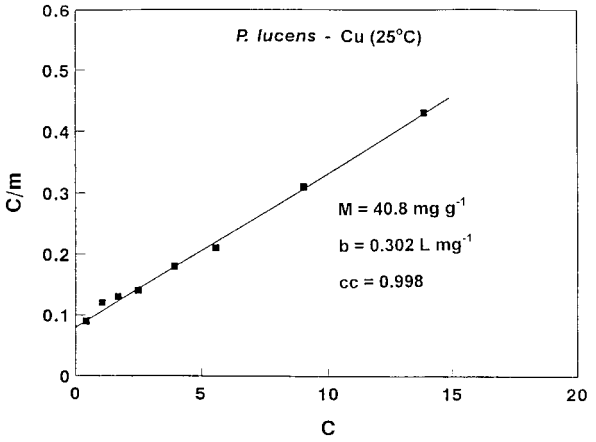


**FIGURE 2. Copper sorption isotherm on *P. lucens* according to Langmuir model.**

$L^{-1}$ aqueous solution (Figure 4). The $Cu^{2+}$ residual species, namely, 0.3 mg $L^{-1}$ for *P. lucens*, 0.4 mg $L^{-1}$ for *S. herzogii*, and 1.3 mg $L^{-1}$ for *E. crassipes*, represents 99.8% of the total species present in solution at pH 5.5. No sorption occurs at very low pH values neither when copper hydrolyses to neutral or negative species. The decrease in uptake capacity for lower pH values (2−3) can be explained because $H^+$ ions were present in high concentration and compete with $Cu^{2+}$ ions for the binding sites.

Figure 5 illustrates a typical breakthrough curve obtained in tests using a laboratory column with *P. lucens,* and Figure 6 shows the sorption capacity for copper uptake as a function of the number of cycles. It can be seen that in the first cycle the copper accumulation was about 40 mg $g^{-1}$ for *P. lucens,* in agreement with the maximum sorption capacity of the biomass. Even after several cycles, the biomass showed a high sorption capacity.

Regeneration of the biosorbents with 0.5% HCl was effective and permitted us to run several sorption−desorption cycles. Moreover, the same acid solution allowed, after each cycle, us to concentrate the copper content in the eluent. Under the experimental conditions, values of the order of 2 g of copper $L^{-1}$ were obtained after 10 cycles. However, after each sorption cycle, the regenerant becomes increasingly weaker, and another eluent solution is required.

Problems of column packing or bed channeling were not observed using particles sized about 4.00 mm. Also, physical degradation of the biomass was not found, even after several sorption−desorption cycles. Acid percolation apparently does not attack the tissues. However, percolation with basic solution (pH above 9.0) promotes degradation of the biosorbents.

The treatment of industrial effluent samples containing residual metal ions by *P. lucens* biomass is shown in Table

**TABLE 4. Ion Exchange Balance after Reaction of *P. lucens* Biomass in the Non-Hydrogen Form with a Copper Solution[a]**

| metallic ion species | initial concn in solution (mg $L^{-1}$) | final concn in solution (mg $L^{-1}$) | control[b] (mg $L^{-1}$) |
|---|---|---|---|
| $Na^+$ | ND[c] | 14.8 | 5.1 |
| $K^+$ | ND | 28.0 | 6.7 |
| $Ca^{2+}$ | ND | 12.5 | 0.2 |
| $Mg^{2+}$ | ND | 12.7 | 0.1 |
| $Fe^{3+}$ | ND | ND | 0.09 |
| $Mn^{2+}$ | ND | 2.3 | ND |
| $Cu^{2+}$ | 103 | 2.5 | ND |
| pH | 4.9 | 5.1 | 7.2 |

[a] 5 g of biosorbent $L^{-1}$, 30-min agitation time. [b] Deionized water and *P. lucens* biomass. [c] ND, not detected.

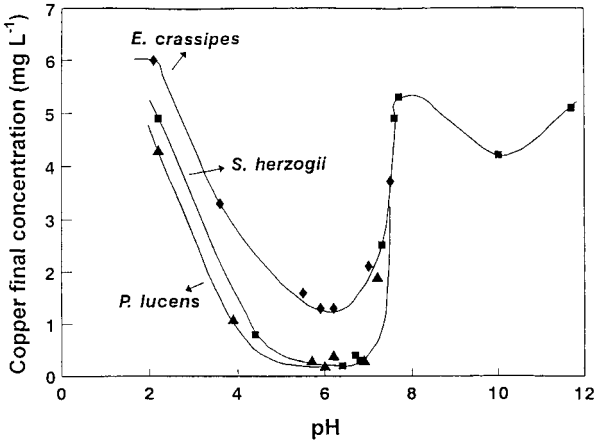


**FIGURE 3. Effect of pH on copper sorption by *P. lucens*, *S. herzogii*, and *E. crassipes* in particle size less than 0.59 mm. Initial Cu(II) concentration of 6.3 mg $L^{-1}$, 30 min agitation time, 2 g of biosorbent $L^{-1}$.**

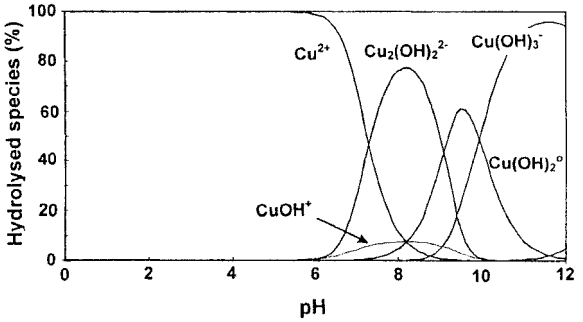


**FIGURE 4. Concentration diagram of copper species in aqueous solution. Copper concentration: $1 \times 10^{-4}$ M (6.3 mg $L^{-1}$). Equilibrium data, taken from Baes and Mesmer (*20*).**

5. The treatment procedure was carried out, monitoring the breakthrough curve of the treated water. The feed was interrupted when the effluent did not attain the local emission limits. The sorption ability of the biomass in the metal uptake in the plating effluent was inferior to the copper leaching plant. This is because in the first one some residual interfering surfactants and metal cyanide complexes are present.

## Discussion

The results obtained clearly demonstrate that the biomass of the nonliving aquatic macrophytes behaves as weak cation exchanger materials. The metal uptake proceeded through exchange with, among other, $Na^+$, $K^+$, $Mg^{2+}$, $Ca^{2+}$, $Fe^{3+}$, and $Mn^{2+}$. Stequiometric relationships obtained confirm that ion exchange is the main mechanism responsible for the ions uptake. This mechanism has also been proposed for the metal

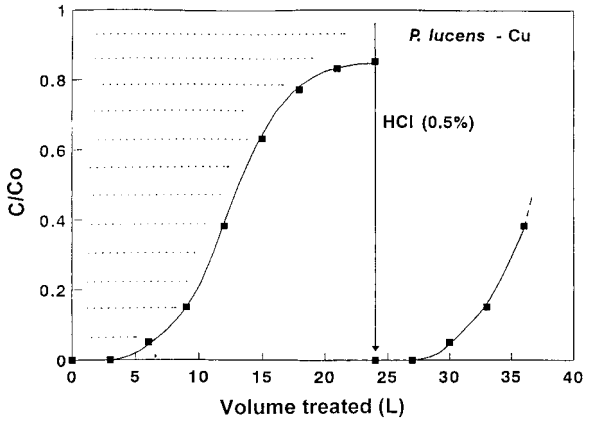


**FIGURE 5. Breaktrough curve obtained in a laboratory column with *P. lucens* in particle size less than 4.0 mm.**

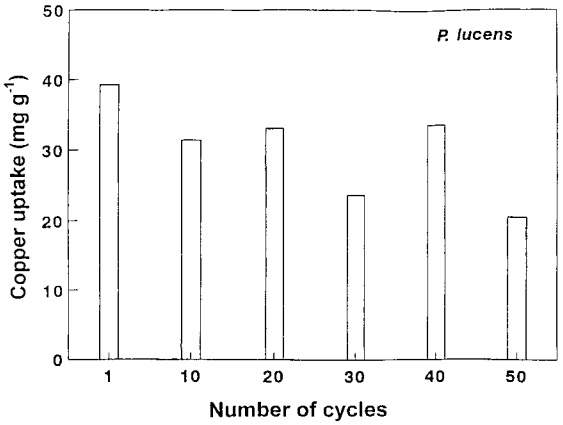


**FIGURE 6. Copper uptake capacity of *P. lucens* as a function of the number of sorption/desorption cycles.**

**TABLE 5. Treatment of Industrial Effluents by Adsorption on *P. lucens* Biomass at pH 5.5 and a Flow Rate of 1.5 bed vol/min[a]**

| | metal contaminants | | |
|---|---|---|---|
| effluent | feed (mg $L^{-1}$) | breakpoint (mg $L^{-1}$) | vol treated (bed vol) |
| copper leaching plant | Cu, 2.0 | Cu, 0.5 | 900 |
| electroplating effluent | Ni, 1.7<br>Cu, 0.1<br>Zn, 2.2 | Ni, 0.9<br>Cu, ND[b]<br>Zn, 1.0 | 200 |

[a] Brazilian emission levels: Ni <1.0 mg $L^{-1}$, Cu <0.5 mg $L^{-1}$, Zn <1.0 mg $L^{-1}$. [b] ND, not detected.

binding properties of Sphagnum mosses (*21*) and marine algae (*22*, *23*).

The main functional group in the ion exchange reactions at neutral values of pH are the carboxyl present on the plant tissues. The carboxyl groups appear to be related to the protein content in the tissues since *P. lucens*, which showed the highest protein content, also presented the highest concentration of carboxyl groups. The metal binding probably occurs with the free carboxyl groups present in the glutamic and aspartic amino acids of the protein chains. However, despite carboxyl groups being mainly responsible for metal complexation by natural organic matter (*24*), biological tissues should have a small amount of nitrogen and sulfur sites that may be important ligand atoms and may also play a role on metal sorption (*8*, *24*).

The sorption capacities were on the order of 1.5 mequiv $g^{-1}$ for *P. lucens*, 0.9 mequiv $g^{-1}$ for *S. herzogii*, and 0.7 mequiv

$g^{-1}$ for *E. crassipes*. The values exceeded many previously reported uptakes for other natural occurring biomass types. Highest sorption capacities with divalent heavy metals have been obtained with native *Sargassun natans* (3.5 mequiv $g^{-1}$) (*23*) and some chemically reinforced biomasses, e.g., the macroalgae *S. fluitans* (4.0 mequiv $g^{-1}$) (*22*) and modified barks (1.6 mequiv $g^{-1}$) (*25*). Compared to commercial resins, the carboxylic ones have an ion exchange capacity of about 8 mequiv $g^{-1}$ (*17*), and the chelating ones have an ion exchange capacity of approximately 4 mequiv $g^{-1}$ (*26*).

The economic success of a biosorbent based on aquatic plant biomass depends to a large extent on the growth rate of the plant. Among all freshwater macrophytes, the floating water hyacinth has the highest growth rate, with a potential yield of about 500 kg $ha^{-1}$ $day^{-1}$ (*27*). Species of the *Salvinia* genera are also aggressive free floating weeds that usually cause problems in waterways of countries outside its native habitat of South America (*28*). In Florida, for instance, because of good climate conditions, biomass productivity of *Salvinia* sp. may reach about 20−120 kg $ha^{-1}$ $day^{-1}$ (*28*). Certain emergent and submerged plants are also quite productive. Typical values of submerged species growth (e.g., *Potamogeton* sp.) are on the order of 30−50 kg $ha^{-1}$ $day^{-1}$ (*29*).

The cost of these products arises mainly from harvesting (relatively cheap), drying, grinding, packing, and transportation. In Brazil, for example, the price of the dried biomass of the *E. crassipes* for methane/alcohol production, cattle feed, and organic fertilizer does not exceed $0.20 (U.S)/kg. This is a very low cost as compared with ion exchange resins, which cost between $5 and $28 (U.S.)/kg.

Thus, it is believed that sorption of diluted heavy metal ions by dried *P. lucens*, *E. crassipes*, or *S. herzogii* appears to be a cheap and efficient alternative to be considered. These plants grow greatly in tropical areas worldwide. After preparation, they can be packed and utilized as such or as filters without the need for immobilization to solve the problems of handling. They can be used in batch reactors, packed bed columns, or fluidized-bed contactors in wastewater treatment plants as a polishing stage.

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