Desalination and Water Treatment
www.deswater.com

doi: 10.1080/19443994.2015.1042061

57 (2016) 11103–11110
May

Taylor & Francis
Taylor & Francis Group

Spirulina platensis as biosorbent of chromium and nickel from industrial effluents

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Received 12 August 2014; Accepted 9 April 2015

ABSTRACT

The time-dependent biosorption capacity of the cyanobacterium *Spirulina platensis* was studied in regards to nickel and chromium removal from the industrial effluents of electroplating units. The elemental content of the *S. platensis* biomass and the metal concentrations of industrial effluents were traced through two analytical techniques, neutron activation analysis and atomic adsorption spectrometry, respectively. A rapid rate of chromium adsorption (initial concentration 9.4 mg/L) was observed within the first 15 min of the reaction. Furthermore, a high amount of iron (48%) and nickel (94%) was removed from the chromium containing industrial effluents by the spirulina biomass. During a 1 h period of the reaction, the biomass of cyanobacterium accumulated from the nickel containing industrial effluents 66% of the initial nickel content (14.1 mg/L), 52% of the initial iron, 30% of the initial zinc, and 50% of the intial barium content. Fourier transform infrared spectroscopy was used to identify functional groups responsible for metal binding.

Keywords: Biosorption; Chromium; Nickel; Neutron activation analysis; FT-IR spectroscopy; *Spirulina platensis*

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1. Introduction

In recent years, there has been increasing interest in cost-effective and environmentally friendly processes for the remediation of metal contaminated waters. As an alternative to electrochemical treatment, ion exchange, precipitation, evaporation, and sorption [1]; biological methods based on effective biosorbents for water remediation such as fungi, bacteria [2–4], cyanobacteria and microalgae [5–7] have seen rapid development.

The use of micro-organisms for the purification of contaminated water has a number of advantages: it is readily available, since micro-organisms are present in unlimited quantities in seas and oceans; it is time and cost-efficient process, due to the rapid kinetics of heavy metal recovery; and it can remove metal ions even when they are present in low concentrations [8,9]. Cyanobacteria and microalgae are of particular interest in the development of new materials with biosorption properties. These organisms accumulate metals from the environment by the passive adsorption of metals on the cell surface, such as the cell wall, membrane, and capsular polysaccharides, or by the formation of complexes with intracellular or exocellular molecules [10–12]. Cyanobacteria play an important role in the treatment of industrial effluents containing heavy metals in concentrations below 100 mg/L, because traditional techniques become inefficient or very expensive when contaminants are present at trace concentrations. Different functional groups: hydroxyl, phosphate, amino, carboxyl, and sulfhydryl associated with cell wall components such as peptidoglycans, teichuronic acids, teichoic acids, polysaccharides, and proteins play an important role in the capture of various metals in the aquatic environment [13]. Major factors that influence the efficiency of rapid biosorption are the initial concentration of the metal, temperature, pH, and biomass concentration in the solution. Temperature does not affect the biosorption if it varies within the range of 20–35˚C, while the pH always affects the chemical form of the metals, the activity of the functional groups of the biomass, and the competition of metal ions for binding sites [13–17].

Numerous investigations have shown that *Spirulina platensis (S. platensis)* can be efficiently applied as bioaccumulator and sorbent of different metals and radioactive ions [18–23]. Despite this, scientific literature is limited to only showing the ability of spirulina to remove metal ions in single- and multi-metal model solutions. *S. platensis* is also widely used as a matrix for pharmaceuticals with some essential elements as well as a biologically active food additive for humans and animals [24].

Chromium, nickel, and their alloys are widely used in various chemical processes due to their resistance to organic and inorganic substances as well as atmospheric agents even at high temperatures. Chromium (VI) has been recognized as one of the most serious pollutants among heavy metals in the environment [25], because it is carcinogenic and may cause skin and respiratory diseases through a diverse range of pathways [1]. Along with chromium, nickel is one of the most well-known human carcinogens. In high concentrations, nickel can cause dermatitis, nausea, vomiting, and behavioral and respiratory problems in addition to cyanosis, gastrointestinal distress, and weakness [26]. Therefore, the removal of chromium and nickel from wastewater is of utmost importance.

The aim of the present work was to study nickel and chromium uptake by cyanobacteria *S. platensis* from chemically complex industrial effluents with the goal of water recycling. Atomic adsorption spectrometry (AAS) was used for the determination of chromium and nickel concentrations in supernatant solutions (industrial effluents). Furthermore, multi-element neutron activation analysis (NAA) was applied to study the elemental content of the biomass after the sorption process. Functional groups responsible for metal binding were determined by Fourier transform infrared spectroscopy (FT-IR).

2. Materials and methods

2.1. Industrial effluents

Two types of industrial effluents were obtained from electroplating units of the Scientific Production Association "Atom", a producer of a large volume of construction steel parts, dedicated tanks and vessels, including those operated under pressure and in aggressive environments (Dubna, Russia): the first one containing chromium in concentration of approximately 9.4 mg/L and the second one containing nickel in concentrations of approximately 14.1 mg/L along with other metals such as barium, cobalt, copper, iron, scandium, strontium, and zinc. Industrial effluents were collected directly after the electroplating process. Current treatment schemes of industrial effluents for metal removal include a complex array of chemical methods.

2.2. Biomass

In the experiments, the *CNM-CB-02* strain of the cyanobacterium *S. platensis* was used. The cultivation of *S. platensis* was carried out in an open-type tank with a volume of 60 L, pH 8–9, and was under constant mixing. The cultivation of the *S. platensis* cells was conducted for 6 d [27]. The biomass of *S. platensis* was collected after 6 d of cultivation and was separated from the nutritive environment by vacuum filtration, washed with distilled water, and repeatedly filtered.

3. Experiments

In the study, two types of experiments were performed with chromium- and nickel-containing industrial effluents, respectively. The conditions of the experiments and the amount of biomass used for the process of metal removal for both experiments were the same. The experiments were performed at room temperature. To determine the contact time required for equilibrium sorption, 10 mL (ρ = 10 g/L) of wet *S.platensis* biomass was added to 100 mL of industrial effluents containing either chromium or nickel ions in 250 mL capacity Erlenmeyer flasks placed on a rotary shaker set at 100 rpm. The dynamics of the adsorption processes was studied during 1 h. The samples were removed at different time intervals (5, 15, 30, and 60 min), and then filtered and dried. All the experiments were conducted in triplicate and the averages of the measurements for each treatment were used. Chromium and nickel content in the supernatant (industrial effluents) solutions was determined by atomic absorption spectrometry (AAS). The obtained biomass (filtered and dried) was packed in polyethylene bags and aluminum cups for NAA.

3.1. Atomic absorption spectrometry

For AAS measurements, the standard solutions with concentration of 1 g/L were diluted and a series of standards of determined metals were prepared. Chromium and nickel concentrations in the filtrate samples were determined on an AAC-spectrometer at a resonance line of 357.9 nm for chromium and of 232.0 nm for nickel, respectively, using an air–acetylene flame. The current strength was 7 mA.

3.2. Neutron activation analysis

To determine the elemental content of the *S. platensis* biomass, NAA at the pulsed fast reactor IBR-2 (FLNP JINR, Dubna) was used. The description of the irradiation channels and the pneumatic transport system REGATA of the IBR-2 can be found elsewhere [28]. To determine short-lived isotopes, samples were irradiated for 3 min under a thermal neutron fluency rate of approximately 1.6×10^{13} n cm^{-2} s^{-1} and measured for 15 min. In the case of long-lived isotopes the samples were irradiated for 4 d under a resonance neutron fluency rate of approximately 3.31×10^{12} n cm^{-2} s^{-1}, repacked and measured using high-purity germanium detectors twice (after 4–5 d and 20–23 d of decay). The chromium content in the samples was determined by γ-line with the energy of 320.1 keV of isotope ^{51}Cr and nickel by γ-line with the energy of 810.7 keV of isotope ^{58}Co through fast neutron reaction ^{58}Ni(n, p)^{58}Co.

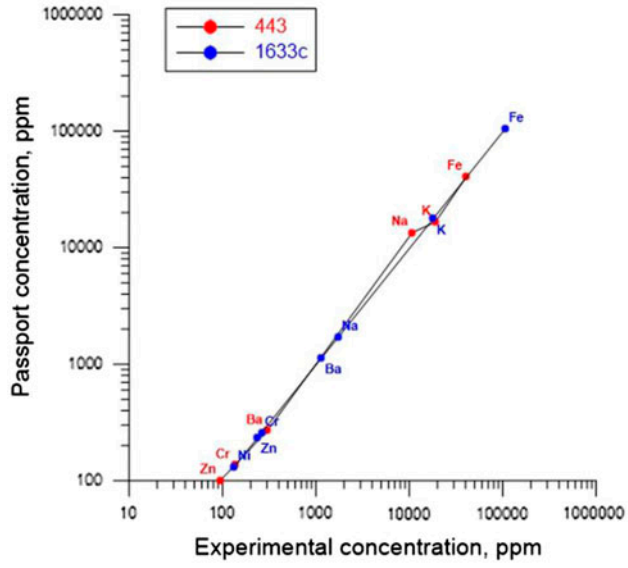

Fig. 1. Comparison of the concentrations obtained experimentally by the NAA and passport data for the used reference materials.

High quality was ensured by the use of the certified reference materials: Trace Elements in Marine Sediment (IAEA 433) and Coal Fly Ash (1633c). The reference materials were irradiated in the same conditions with samples. The correlation between the certified (recommended) values of concentrations and the experimentally obtained ones is presented in Fig. 1. Good agreement was obtained between the experimental and certified values.

The NAA data processing and determination of element concentrations were performed using the software developed in FLNP JINR [29].

3.3. Fourier Transform Infrared Spectroscopy

FT-IR spectroscopy was used to confirm the presence of the functional groups in the samples of *S. platensis* and to observe the chemical modification after heavy metal adsorption. Infrared spectra were recorded in the 4,000−600 cm^{-1} region using a Perkin Elmer Spectrum 100 FT-IR spectrometer.

4. Results and discussion

An important step going forward in the study of metal biosorption is the determination of the optimal time required for the *S. platensis* biomass to bind the maximum amount of chromium and nickel ions. It is well known that metal ion uptake by micro-organisms

is a two-step process. The rapid initial uptake is associated with extracellular sorption and the slower one is due to intracellular accumulation [30].

The NAA results (Fig. 2) showed the rapid uptake of chromium by biomass in first 15 min of reaction. The amount of chromium in the biomass after 1 h of interaction was 60 times higher than in the native biomass (initial concentration of chromium in biomass was 9 μg/g). According to AAS data (Fig. 2) during 60 min of reaction chromium was completely removed from industrial effluents.

In the case of the industrial effluents containing nickel, the results of AAS show that during 60 min of reaction 66% of nickel were removed from industrial effluents. In the spirulina biomass, the nickel content increased from 5 to 625 μg/g (Fig. 3).

The lower nickel removal can be explained by:

(1) its higher than chromium concentration in the industrial effluents. At lower concentrations, metal ions present in the industrial effluents easily interact with the binding sites, thus facilitating higher adsorption. At higher concentrations, adsorption is reduced due to the saturation of binding sites [31].
(2) the presence of other cations in the wastewater, which compete with nickel for binding sites [32]. Industrial effluents from galvanic industry beside chromium and nickel contained other metal ions such as barium, cobalt, copper, iron, scandium, strontium, and zinc can influence the *S. platensis* biomass biosorptive capacity. NAA data showed that besides nickel, a high amount of iron, zinc, and barium was accumulated by the biomass *S. platensis* from the industrial effluents containing nickel (Fig. 4). After 1 h of interaction, the spirulina biomass accumulated the amount of iron and barium that exceeds their concentrations in the control biomass by a factor of 2, and zinc by a factor of 1.5. Corder and Reeves [32] have shown that biosorption of nickel by *Anabaena flos-aquae* from a complex of industrial effluents was considerably lower than its sorption from the industrial effluents containing only nickel.

The high concentration of iron was also estimated in the industrial effluents containing chromium (Fig. 5). In the Cr-loaded biomass, the concentration of iron increased by twofold and nickel increased by 45 times in comparison with the native biomass. The copper content in the native biomass was below the detection limit for analysis, but its concentration achieved the value of 250 μg/g after 1 h of interaction.

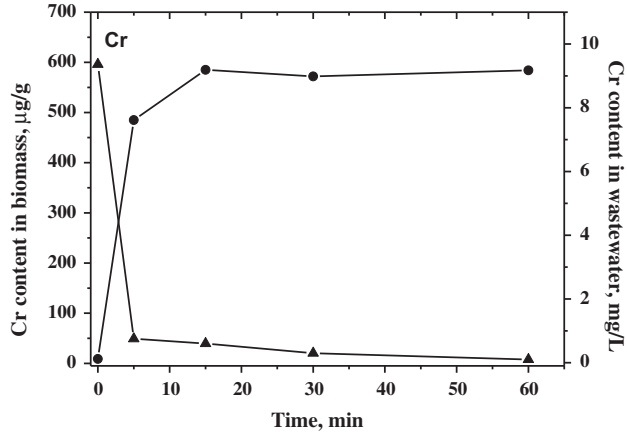

Fig. 2. Chromium content in the *S. platensis* biomass and in the industrial effluents versus the contact time.

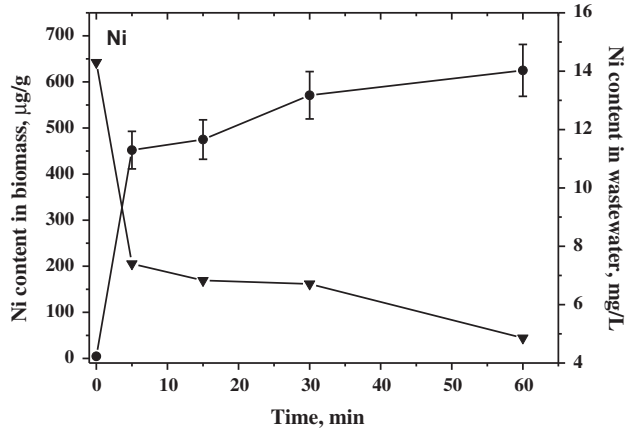

Fig. 3. Nickel content in the *S. platensis* biomass and in the industrial effluents versus the contact time.

Metal adsorption is also a pH-dependent process; the pH of the studied industrial effluents was around 5. A medium pH affects the solubility of metals and the ionization state of the functional groups such as carboxylate, phosphate, and amino groups of the cell wall. The carboxylate and phosphate groups carry negative charges that allow the cell wall components to be potent scavengers of cations [33]. The optimal pH for adsorption of Cr by *Pantoea* sp. TEM18 was 3, for *Aspergillus* sp. 5, and for *Micrococcus* sp. 7 [34]. The optimum pH value for nickel adsorption is 4.5 for *Acinetobacter baumannii* and *Pseudomonas aeruginosa* [35], 6.7 for *Bacillus subtilis* 10 M and 6 for *Codium vermilara* [36].

At low pH values, protons occupy most of the biosorption sites on the algal surface, and the

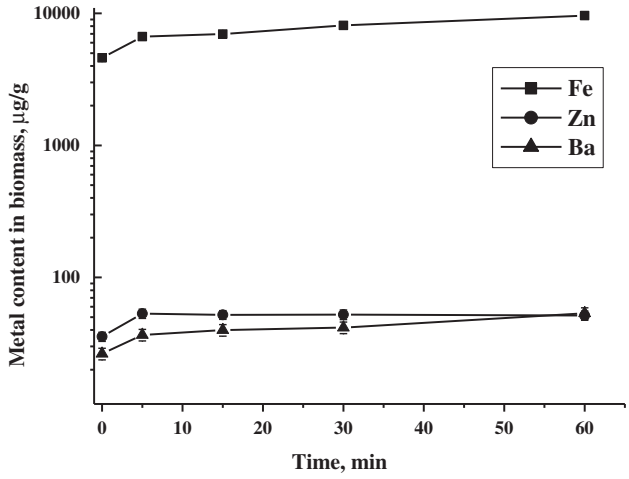

Fig. 4. Fe, Zn, and Ba concentrations in *S. platensis* biomass as a function of the contact time with the industrial effluents containing nickel.

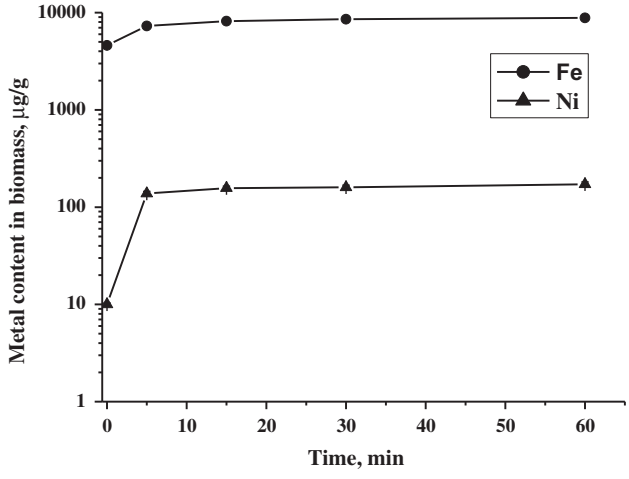

Fig. 5. Fe and Ni concentrations in *S. platensis* biomass as a function of the contact time with the industrial effluents containing chromium.

biosorption of cations is lower. At pH around 5, the biomass surface is more negatively charged, and the sorption is higher for metal ions with a positive charge as Nickel(II). At a pH value higher than 5, the removal of Ni(II) decreases due to the formation of $Ni(OH)_2$ [34,37]. At an acidic pH, the predominant species of Cr(VI) are $Cr_2O_7^{2-}$, $HCrO_4^-$, and $Cr_2O_4^{2-}$ and the surface of the sorbent becomes protonated and attracts anionic species of Cr(VI) [37]. High iron accumulation can be explained by its almost identical uptake within a rather wide range of pH from 1 to 7.5.

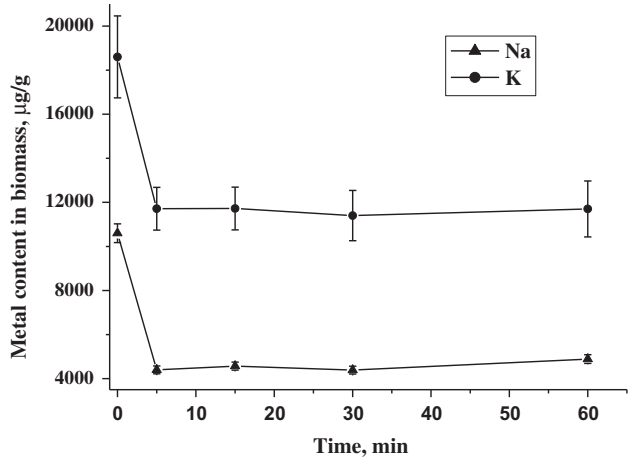

Fig. 6. Change of Na and K concentration in *S. platensis* biomass as a function of the contact time with the industrial effluents containing chromium.

Metal binding to a microbial surface occurs through ion exchange, binding to functional groups, complexation, and microprecipitation among others. The release of sodium and potassium ions from cells (Fig. 6) responsible for the coherence of the cell membrane can be explained by their replacement with other metals. It is also an indicator of the damage of membrane permeability. The same results were obtained for *S. platensis* biomass after the interaction with the industrial effluents containing nickel.

To understand better the nature of functional groups responsible for the chromium and nickel biosorption, FT-IR analysis was performed. The FT-IR spectrum of control biomass (Fig. 7(a)) showed several intense characteristic bands at 1,055.0 and 1,397.9 cm^{-1} due to –OH stretching vibration and vibration of $–NH_2$ are located at 3,277.0 cm^{-1}. The presence of $S\text{-}CH_2$, $NHC(O)_{amid}$, C=O, P=O, and $N\text{-}CH_2$ groups is confirmed by stretching vibration at 701.66, 1,235.8, 1,516.8, 1,625.0, and 2,924.9 cm^{-1}, respectively.

Spectral analysis of Cr-loaded biomass (Fig. 7(b)) showed that OH groups were involved in the binding with chromium, after interaction with wastewater their adsorption bands were shifted 7 and 4 cm^{-1}, respectively. The decrease in the intensity of the adsorption bands of NH_2, P=O, and C=O groups likewise indicates their interaction with chromium ions.

Shift of the position of 1,064.3 cm^{-1}, 1,392.2 cm^{-1}, and 1,238.8 cm^{-1} peaks indicates binding of metals with OH and $NHC(O)_{amid}$ groups in Ni-loaded biomass (Fig. 7(c)).

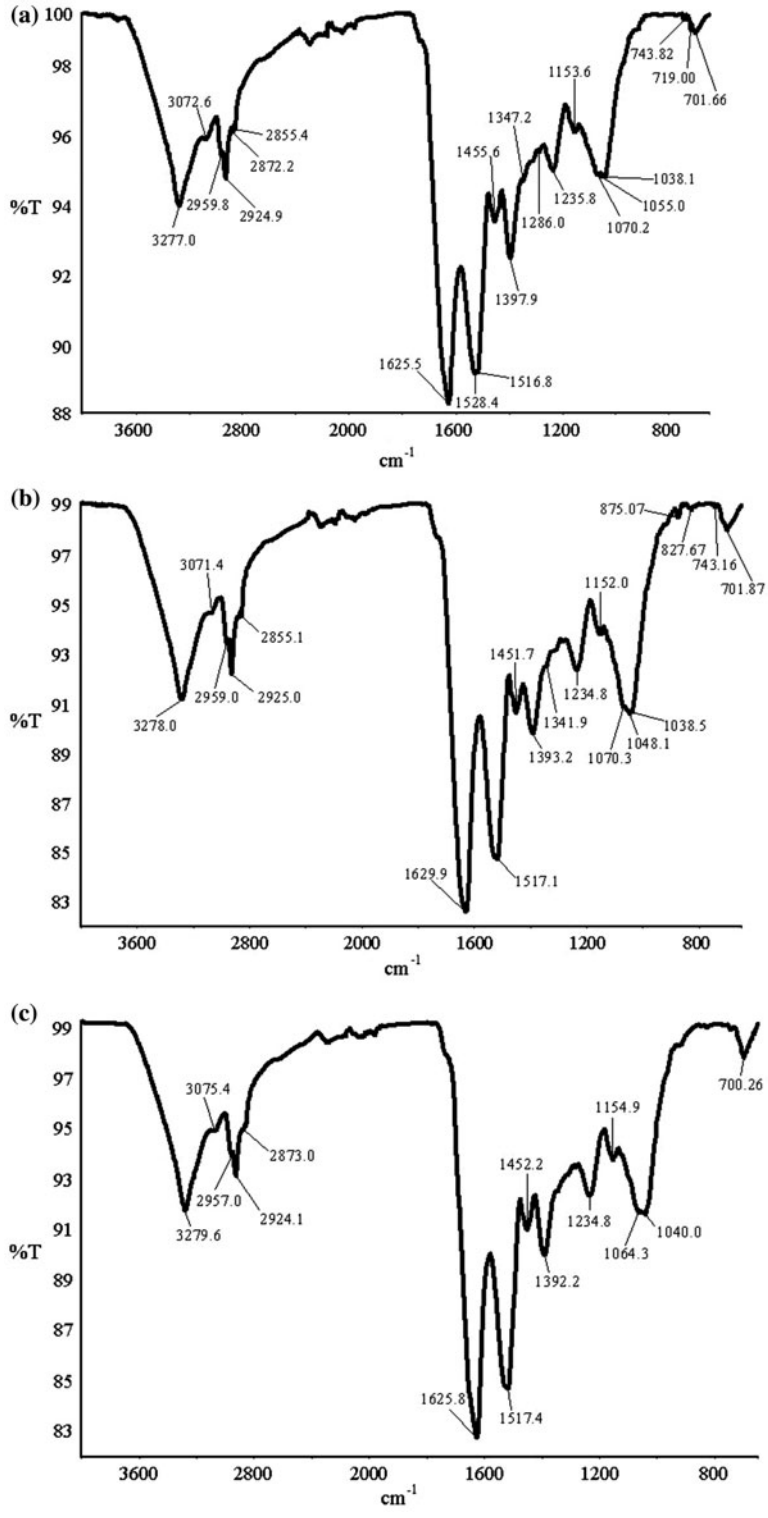

Fig. 7. FT-IR spectra of *S. platensis* biomass: (a) control; (b) Cr-loaded; (c) Ni- loaded.

5. Conclusions

The results of the study demonstrate that *S. platensis* biomass has a great potential as an efficient biosorbent of chromium and nickel from industrial effluents. The obtained data displays high chromium biosorption. Since *S. platensis* biomass accumulates other metals present in industrial effluents, there is the possibility that it can be exploited for multi-metal purification. A decrease in Na and K concentrations indicates the destruction of biomass in the process of metal accumulation. FT-IR spectra have revealed that metal removal takes place through binding to OH, C=O, NH_2, P=O, and $NHC(O)_{amid}$ groups. The biosorption capacity of biomass *S. platensis* serves as a basis for the development of green technology for environmental remediation.

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