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# LITHIUM BIOSORPTION BY Arthrospira (Spirulina) PLATENSIS BIOMASS

**Abstract:** The biosorption of lithium from batch systems by *Arthrospira* (*Spirulina*) *platensis* biomass was studied. Adsorption capacity of the biosorbent was investigated as a function of contact time, initial metals concentration and pH values. Lithium content in biomass was determined using Proton Induced Gamma Emission technique. The ability of spirulina biomass for lithium biosorption showed a maximum at the pH = 11. Equilibrium data fitted well with the Langmuir model with maximum adsorption capacity of 1.75 mg/g, while the kinetic data were best described using the pseudo second-order kinetic model. The IR spectrum of the Li-loaded biomass revealed that lithium ions could be primarily bind to –OH, –COOH, –NH, –NH<sub>2</sub>, and –NH<sub>3</sub> groups present on biosorbent surface. *Arthrospira platensis* biomass could be applied as environmentally friendly sorbent for lithium removal from wastewater.

Keywords: biosorption, lithium, Arthrospira platensis, PIGE technique, FT-IR spectroscopy

# Introduction

Nowadays, the demand for lithium has been increasing dramatically due to its wide application as raw material for large-capacity rechargeable batteries, light aircraft alloys, and future nuclear fusion fuel [1]. It is also applied for production of heat-resistant ceramics, and pharmaceuticals [2]. Lithium batteries are and will also continue to be in demand for powering all electric and hybrid vehicles. Increase use of lithium batteries doubtless will lead to formation of large amount of solid waste, containing lithium amount with other heavy metals [3]. Lithium is now recovered from mines and salt lakes, which contain about 17 Tg (million tonnes) of lithium in total [4].

For lithium removal from solutions are used several traditional techniques such as solvent extraction, ion-exchange, precipitation, membrane processes, and adsorption [1, 2]. However, industrial scale use of ion-exchange resins is limited due to use of toxic chemical that may lead to secondary environment pollution [2]. The conventional techniques for treatment and recycling of metal-bearing wastes, mainly pyro-metallurgy, hydrometallurgy or combination both of them requires high capital investment and can lead to second pollution as well.

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In this case, interest for lithium removal and bioleaching represents biological methods, are considered as one of the most promising and fast developing technologies. Literature search show just several example of microorganism use for lithium recovery. Tsuruta [2] tested 70 strains of microorganisms for their ability to accumulate lithium. Certain gram-positive bacteria were found to have an extremely high ability to accumulate lithium, particularly *Arthrobacter nicotianae*, which can accumulate about 126 µmol Li/g (d.wt.). Marcincakova et al. [3] used consortia of acidophilic bacteria of *Acidithiobacillus ferrooxidans* and *Acidithiobacillus thiooxidans* for lithium bioleaching from spent lithium-ion batteries two different media. In the rich nutrient medium the overall lithium bioleaching efficiency was 80 %, whereas in the low nutrient environment only 35 %. Kurmiawan and Yamamoto [5] demonstrated applicability of biofilm matrices of natural microbial consortiums collected from Lake Biwa, Japan for lithium accumulation.

Cyanobacteria is a group of microorganisms which are widely used in removal of metal ions such from batch solution as well as and industrial effluents [6-14].

The aim of the present study was to examine the efficiency of *Arthrospira (Spirulina)* platensis biomass as a biosorbent for removal of lithium ions from batch solution using Proton Induced Gamma Emission (PIGE) technique. Equilibrium and kinetic studies were performed to describe the adsorption process. Functional groups responsible for metal binding were determined by Fourier transform infrared spectroscopy.

# **Experimental**

#### Reagents and materials

All the chemicals used for biosorption experiments were purchased from Sigma-Aldrich and were of analytical grade.

#### **Biosorbent**

Arthrospira platensis (A. platensis) biomass purchased from "Biosolar MSU" company (Moscow, Russia) was dried in an oven at 80 °C for 24 h. Then the biomass was homogenized in a homogenizer at 600 rpm for 10 min and afterwards used in the experiments.

## **Batch experiments**

The experiments were conducted in 100 cm<sup>3</sup> Erlenmeyer flasks containing 50 cm<sup>3</sup> of lithium synthetic solutions. The flasks were shaken on a shaker incubator at a constant rate of 120 rpm. To investigate the effect of pH, initial metal concentration and contact time, different pH (2-11), initial metal concentrations (10-100 mg/dm<sup>3</sup>), time (5, 15, 30, 45 min) were used. After experiment biomass was removed by filtration, dried till the constant weight and used for further analysis.

The experiments were carried out in triplicate (n = 3) to enhance reproducibility and the mean of the quantitative results were used for further calculations. The value of standard deviation for a sample was not more than 5 %.

#### Methods

Proton Induced Gamma Emission (PIGE) technique was used for a quantitative biosorbent analysis.

### Sample preparation for PIGE

Dry powder samples were ground in agate mortar, then prepared as pellets (thick targets) of 1 cm diameter and 1 mm thickness using hydraulic press. As comparator standards for lithium determination, LiNH<sub>2</sub> powder was similarly prepared as pellets (relative standardization).

#### **Experimental set-up for PIGE**

PIGE experiments were performed in a dedicated to Ion Beam analysis (IBA) reaction chamber at the 3 MV Tandetron of IFIN-HH in Bucharest-Magurele, Romania, using a 3 MeV proton beam normal to the target support (Fig. 2b), in vacuum (beam diameter on target of about 2 mm) [15]. Characteristic gamma-rays from  $^7\text{Li}(p,p'\gamma)^7\text{Li}$  and  $^7\text{Li}(p,n\gamma)^7\text{Be}$  nuclear reactions (477.6 and 429.1 keV, respectively) were measured using a spectrometric chain with GEM10P4-70 High Purity Germanium (HPGe) detector (1.75 keV energy resolution at 1.33 keV of  $^{60}\text{Co}$ ), oriented at 45° with respect to the target and beam direction. Beam charge on target of 22-30  $\mu\text{C}$  and counting time around 3000 s were employed. An example of PIGE spectrum of Li-loaded biomass is shown in Figure 1.

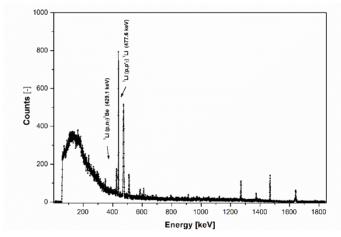


Fig. 1. Typical PIGE spectrum of Li-loaded Arthrospira platensis biomass ( $E_p = 3$ MeV,  $t_c = 2700$  s,  $Q = 21.71 \mu$ C)

Quality assurance. To provide quality control of PIGE results, high purity chemical compound LiNH<sub>2</sub> were used for PIGE calibration and/or quantitative standardization. The difference between certified and measured content of elements of the certified material varied between 3 and 7 %.

Fourier-transform infrared spectroscopy was used to confirm the presence of the functional groups in the samples of A. platensis and to observe the chemical modification after nickel biosorption. Infrared spectra were recorded in the range of 4,000-400 cm<sup>-1</sup> using a Bruker Alpha Platinum-ATR spectrometer (Bruker Optics, Ettingen, Germany).

#### **Results and discussion**

#### Effect of pH on biosorption

The initial pH value of adsorption solution is an important factor, which must be considered during sorption studies [4]. In present study, the effect of pH was studied at pH range 2-11. Obtained data are presented in Figure 2. As it can be seen from Figure 2 increase of pH value lead to the increase of biomass sorption capacity. The lithium sorption reached the maximum (493  $\mu$ g/g) at pH 11. Obtained results are confirmed by Park et al. [16], who studied the effect of pH in the range 2-12 on lithium sorption on  $\lambda$ -MnO<sub>2</sub> adsorbents and showed the continuous increases of lithium biosorption with pH increases. Wang et al. [4] showed that uptake of Li<sup>+</sup> by ion-sieve was favourable at pH values 10-12. Even in our previous studies, it was shown that optimal pH range for metal cationic species sorption is 4.0-6.0 [10, 11, 17], in case of lithium sorption was more favourable at high pH values.

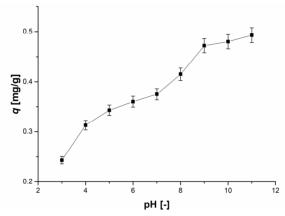


Fig. 2. Removal of lithium ions at different initial pH (at T = 20 °C;  $C_i = 10$  mg/dm<sup>3</sup>; sorbent dosage 10 g/dm<sup>3</sup>; adsorption time 2 h)

Wang et al. [4] concluded that lithium uptake should be carried out in stronger basic solution or rather in basic buffer solution. Authors suggested that in neutral or weak alkaline solutions due to ion-exchange reaction between  $Li^+$  and  $H^+$  the solution pH could be changed to acid zone. At low pH values completion of protons and lithium ions may take place that will result in incomplete lithium removal. To support Wang et al. [4] suggestion the pH of the sorbent-sorbate solution was measured at the beginning of the experiment and after two hours. For example, in case than initial pH value was 11 after two hours experiment it became 5.6. In Tsuruta [2] study it was found that lithium accumulation by the microorganisms was maximum at pH = 5.8.

#### Effect of time and kinetics of sorption

The evolution of the uptake data of lithium as function of time is illustrated in Figure 3. A rapid increase of lithium sorption in the first 45 min of interaction was observed after that the equilibrium was reached. During 30 min 90 % of lithium was removed from solution by spirulina biomass. In first 5 min of interaction lithium content in biomass

increases by 53 % (in control biomass lithium was not detected). Tsuruta [2] have showed that maximum amount of lithium was accumulated by the *A. nicotianae* cells rapidly increased in the first 5 min of biomass-solution interaction. Kurmiawan and Yamamoto [5] demonstrated that maximum amount of lithium was sorbed by biofilm matrix after 1-min biosorption.

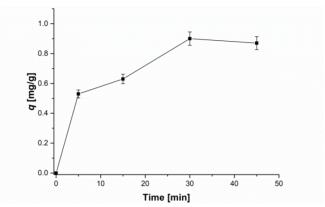


Fig. 3. Effect of contact time on the sorption of nickel ions by A. platensis biomass (at T = 20 °C;  $C_i = 10 \text{ mg/dm}^3$ ; pH = 11; sorbent dosage 10 g/dm<sup>3</sup>)

Lagergren's pseudo-first order and pseudo-second order models of Ho and McKay [18] were used to fit the experimental data.

Lagergren's pseudo-first order model is expressed as follows:

$$\log(q_e - q) = \log q_e - \frac{K_a}{2.303}t\tag{1}$$

where q and  $q_e$  are the adsorbed amounts [mg/g] at time t [min] and at equilibrium time, respectively,  $K_a$  [min<sup>-1</sup>] is the rate constant of the first order biosorption.

Pseudo-second order model of Ho and McKay:

$$\frac{t}{q} = \frac{1}{K_h q_e^2} + \frac{t}{q_e} \tag{2}$$

where q and  $q_e$  are the adsorbed amounts [ $\mu$ g/g] at time t [min] and at equilibrium time, respectively,  $K_b$  [g/(mg·min)] is the rate constant of the second-order biosorption.

Pseudo-first order model was not applicable to describe experimental data, correlation coefficient was 0.97 (data not shown). The pseudo-second order biosorption rate constant,  $K_b$  and  $q_e$  values were determined from the slope and intercept of the plot of  $t/q_t$  against time, t (Fig. 4) and obtained parameters are presented in Table 1. Fitted pseudo-second order model kinetic parameters for the removal of lithium ions by spirulina biomass are presented in Table 1. The pseudo-second order model, based on the assumption that the rate-limiting step is chemical sorption involving valency forces through sharing or exchange of electrons between sorbent and sorbate [19, 20].

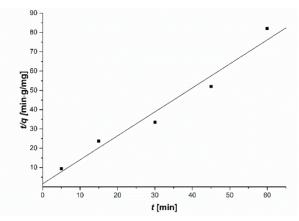


Fig. 4. The pseudo-second order plot of kinetic study of lithium biosorption on A. platensis

The pseudo-second order model parameters

0.9

Pseudo-second order  $q_{e(exp)}[mg/g]$   $q_{e(cal)}[mg/g]$   $K_b[g/(mg\cdot min)]$   $R^2[-]$ 

1.97

Table 1

0.99

# Effect of concentration and isotherm modelling

0.87

Increase of metal concentration in solution was in direct ration with its uptake by biomass. The maximum achieved adsorption capacity was 1.3 mg Li/g of biomass.

Two models: Langmuir and Freundlich were used to describe the experimental data:

The Langmuir model expressed as:

 $C_e \, [\text{mg/dm}^3]$ 

10

$$\frac{1}{q_e} = \frac{1}{q_{max}} + \frac{1}{bq_{max}C_e} \tag{3}$$

where  $q_e$  is the amount of metal adsorbed per unit weight of adsorbent at equilibrium [mg/g],  $q_{max}$  is the maximum metal uptake per unit mass of the adsorbent (mg/g], b is the Langmuir constant [dm³/mg], related to the energy of sorption, which quantitatively reflects the affinity between the sorbent and the sorbate and  $C_e$  is the equilibrium concentration of adsorbate [mg/dm³].

The general Freundlich equation is written as follows:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \tag{4}$$

where  $K_F$  and 1/n are Freundlich constants, associated with adsorption capacity and adsorption intensity, respectively.

The isotherm parameters have been calculated from linearized plots corresponding to each isotherm model (Fig. 5) and are summarized in Table 2.

Table 2

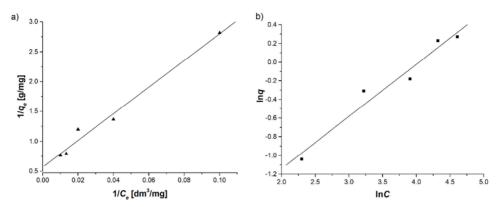


Fig. 5. a) Langmuir and b) Freundlich isotherm models for dry biomass

Isotherm parameters for the biosorption of lithium ions on *A. platensis* biomass

Langmuir model		Freundlich model	
$R^2$ [-]	0.99	$R^2$ [-]	0.97
$q_{max}$ [mg/g]	1.75	$K_F$ [mg/g]	0.1
b [dm³/mg]	0.015	n [-]	1.78

A basic assumption of the Langmuir theory is that sorption takes place at specific homogeneous sites within the sorbent. It is suggested that once a metal ion occupies a site, no further sorption can take place at that site [20]. The value of correlation experiment indicate that sorption data follows the Langmuir sorption isotherm. Correlation coefficient for Freundlich model was lower than that obtained for Langmuir model. Experimentally obtained maximum q value was comparable to the maximum adsorption obtained from the Langmuir isotherm. This suggest that lithium is adsorbed in the form of monolayer coverage on the surface of the prepared adsorbent [21].

# Mechanisms of lithium biosorption

The mechanism of metal biosorption includes adsorption on surface, binding to functional groups, diffusion through pores and ion exchange [22]. In order to confirm participation of the functional groups in lithium biosorption the biomass was analysed by FTIR spectroscopy. Spectrum of control biomass shows several major intense bands, around wavenumbers 3,282; 2,926; 1,634; 1,538; 1,454; 1,392; and 1,056 cm<sup>-1</sup> (Fig. 6). The strong boarded peak at wavenumber 3,282 cm<sup>-1</sup> could be attributed to hydroxyl (–OH) and amine (–NH) functional groups [22-24]. The presence of methyl (–CH) stretching vibrations could be confirmed by the adsorption peak at wavenumber 2,926 cm<sup>-1</sup> [24]. The adsorption peaks in the region at wavenumbers 1,750-1,300 cm<sup>-1</sup> could be assigned to –CO stretches of aldehydes, ketones, and carboxylate [22, 23]. In addition, –C–O, –C–C, and –C–OH stretching vibrations could be found at the adsorption peaks of the 1,250-1,000 cm<sup>-1</sup> region [22, 25]. The strong bands at wavenumbers 3,282; 1,634; 1,538; and 1,242 cm<sup>-1</sup> could be also corresponding to the amide I–III bands of polypeptide or proteins, respectively [26]. The insignificantly deformations in the region 900-500 cm<sup>-1</sup> could be attributed to –P–O, and aromatic –CH stretching vibrations [22, 25].

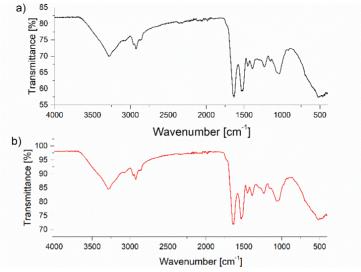


Fig. 6. FT-IR spectra of A. platensis biomass: a) control and b) Li-loaded

The IR spectrum of the Li-loaded spirulina biomass revealed that the hydroxyl (3,278 and 1,633 cm<sup>-1</sup>), amine (3,278 cm<sup>-1</sup>), carboxyl (1,750-1,300 and 1,250-1,000 cm<sup>-1</sup>), and phosphate (900-500 cm<sup>-1</sup>) functional groups were slightly shifted in comparison with their positions showed on Figure 6. It was caused by Li ions binding to above mentioned groups. In Yun and Volensky study [27] was shown that carboxyl and phosphate groups play important role in lithium ions binding.

Ion-exchange can be considered as another mechanism of lithium biosorption. Since lithium belongs to the group of alkali metals together with sodium and potassium, it can replace these elements due to similar structures [28].

# Conclusion

The potential of *Arthrospira platensis* biomass application for lithium ions removal from both batch solutions was evaluated. The maximum biosorption capacity of lithium 1.75 mg/g was achieved at pH = 11.0 and sorbent dosage 10 g/dm<sup>3</sup>. The equilibrium data were well fitted by Langmuir adsorption isotherm model ( $R^2 = 0.99$ ), while the pseudo-second order kinetic model ( $R^2 > 0.99$ ) was found to describe better the kinetic data. The FTIR analysis showed that OH, -COOH, -NH, -NH<sub>2</sub>, and -NH<sub>3</sub> were mainly involved in lithium ions binding. Thus, metal trapping to functional groups and ion-exchange can be proposed as main mechanism of lithium biosorption by *Arthrospira platensis* biomass. Obtained results indicated that *Arthrospira platensis* biomass can be used as a cheap and efficient sorbent for lithium removal from wastewater and wastewater post-treatment.

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