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Biosorption of lead ions by cyanobacteria *Spirulina platensis*: kinetics, equilibrium and thermodynamic study

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Abstract

The potential use of dry *Spirulina platensis* biomass to remove lead ions from aqueous solution was investigated. Effects of various parameters such as contact time, temperature, dosage of biosorbent, initial pH, and initial concentration of lead were investigated in the batch adsorption mode. The highest lead removal of 5.7 mg/g was obtained at pH 5, biomass dosage of 0.5 g, initial lead concentration of 60 mg/L. The Langmuir and Freundlich models fit the experimental data ($R^2 > 0.99$), while the kinetic data was best described using the pseudo second-order kinetic model ($R^2 > 0.99$). FTIR spectra indicated that the metal removal takes place through binding to OH, C=O and P=O groups. Lead was efficiently recovered from biomass by mineral acids, while using CH_3COOH and NaOH as eluents the biomass maintained high biosorption capacity during three cycles. This study demonstrates the potential of using *Spirulina platensis* as biosorbent to remove lead from industrial wastewater.

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Introduction

Among the metals, lead is widely used in industry and its release in water bodies may cause serious ecological and human health consequences. In addition to such major lead sources as gasoline vehicles, lead is used as an industrial raw material for storage battery manufacturing, printing, pigments, fuels, photographic materials and explosive manufacturing (Jalali *et al.* 2002; Bahadir *et al.* 2007; Miranda *et al.* 2013). Traditional technologies presently used for metal removal, precipitation, chemical absorption, oxidation-reduction, use of ion-exchange resins, have significant disadvantages, including incomplete metal removal, high cost, generation of toxic sludge or other waste products that require disposal (Bahadir *et al.* 2007; Chen *et al.* 2011). Biosorption is one of the most promising technologies involved in the removal of toxic metal, which allows reducing metal ion concentrations to environmentally acceptable levels. The biosorption process includes physicochemical interactions between metal ions and several anionic ligands present on the biomass like carboxyl, phosphoryl, carbonyl and sulfhydryl

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(Jalali *et al.* 2002). The efficiency of metal biosorption is strongly dependent on experiment parameters such as: pH, temperature, and the concentration of sorbate and sorbent (Chojnacka 2010). Various materials of biological origin, including bacteria, cyanobacteria, fungi and yeasts, can be used for toxic metals biosorption (Bahadir *et al.* 2007; Bhatnagar *et al.* 2010; Miranda *et al.* 2013; Kaduková and Horváthová 2012; Sadovsky *et al.* 2016) *Spirulina* biomass, due to its relatively high sorption capacity and low cost, was studied in the removal of a wide range of toxic metals (Chen and Pan 2005; Vannela and Verma 2006; Lodi *et al.* 2008; Zinicovscaia *et al.* 2016). Gong *et al.* (2006) investigated the effects of operational conditions (pH, contact time, biomass concentration time of interaction) on lead biosorption by intact and pretreated biomass of *Spirulina maxima*. Al-Homaidan *et al.* (2016) studied the effect of the same parameters on the lead removal by non-living *Spirulina platensis* biomass.

The objective of the present study was to examine the effect of different operational parameters on the efficiency of lead biosorption by dry spirulina biomass. Equilibrium, kinetic and thermodynamic studies, not implemented in the previously published work, were performed to describe the adsorption process. Functional groups responsible for metal binding were determined by Fourier transform infrared spectroscopy. The recovery of bound lead by different type of desorbents was measured.

Experimental

Reagents and materials

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

Preparation of adsorbent

Spirulina platensis (*S. platensis*) biomass purchased from "Biosolar MSU" company 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.

Experiment

The influence of pH was studied using lead nitrate solution with an initial concentration of 10 mg Pb/L and pH values varying between 2.0 and 7. The initial solution was adjusted to the desired pH with diluted or concentrated HNO_3 and NaOH solutions before being mixed with the biosorbent. The change in the working volume due to the addition of HNO_3 and NaOH was negligible in this experiment. The effect of biosorbent dosage on batch experiments was examined by varying its weight from 0.1 to 1 g. For the isotherm study, 3 – 400 mg Pb/L was mixed with 0.5 g biomass at pH 5 and shaken at 200 rpm for 1 h. To determine the contact time required for the sorption equilibrium experiments, samples were withdrawn at predetermined time intervals (3, 5, 7, 10, 15, 30, 60 and 120 min). All the experiments were carried out under ambient conditions at 20 °C, except for the thermodynamic studies at temperature 20, 30, 40 and 50 °C, respectively. In all experiments the working volume was 50 mL.

After experiment, biosorbent was separated by centrifugation and the concentration of metal ion in solution was analyzed by atomic adsorption spectrometry. The metal uptake q (mg Pb/g of biosorbent) was calculated from the mass balance using the following equation:

$$q = \frac{(C_i - C_f)V}{m}$$

where q is the amount of metal ions adsorbed on the biosorbent, mg/g; V is the volume of solution, ml; C_i is the initial concentration of metal in mg/L, C_f is the final metal concentration in the solution, mg/L, and m is the mass of sorbent, g.

Desorption experiment

In desorption experiments *S. platensis* biomass was firstly exposed to 30 mg Pb/L solution at optimum pH conditions. Next, the biomass was separated from the solution by centrifugation. The obtained Pb-loaded biosorbent was then brought in contact with 50 mL of 0.1 M HNO_3, 0.1 M HCl, 0.1 M CH_3COOH and 0.1 M NaOH for 1 h on an orbital shaker at 200 rpm. The procedure was repeated for three times.

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Atomic adsorption spectrometry

Lead content in solution was determined in propane flame on KVANT-2a spectrophotometer at a resonance line of 217.0 nm. The strength of the current was 16 mA.

Fourier Transform Infrared Spectroscopy (FT-IR)

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 lead biosorption. Infrared spectra were recorded in the 4 000 − 600 cm^{-1} region using a Perkin Elmer Spectrum 100 FT-IR spectrometer.

Results and Discussion

Effect of pH value on biosorption

The pH value plays an important role in biosorption process, because it influences the speciation of metal in the aqueous solution, the ionization of surface functional groups, and the binding sites on the surface of the biomass (Amini *et al.* 2013). In the present study, the effect of initial solution pH was examined in the range 2.0 – 7.0, and the obtained results are presented in Fig. 1. The equilibrium sorption capacity had a minimum at pH 2 (52%). At pH 2.0, protons, due to their high concentration occupy most of the active sites on the biosorbent surface, thus less lead ions could be adsorbed. The maximum lead biosorption by the biomass (0.8 mg/g) was observed at pH 3 – 5, corresponding to 88 – 89% removal efficiency. There have been similar observations for lead biosorption by *Spirulina* sp. at pH 4 (Aneja *et al.* 2010). At pH values higher than 5.0, a slow decrease of biosorption parameters was observed. The speciation of lead is pH dependent. At pH of 2 – 6 predominant sorbing forms of lead are Pb^{2+} and $PbOH^{+}$, while above pH of 6, it is hydrolyzed to $PbOH^{+}$ and $Pb(OH)_2$ (Qaiser *et al.* 2009). This was the reason for higher removal of lead in the pH range of 3 – 5. Therefore, pH 5.0 was considered as the optimum pH and used in further experiments.

Optimal pH value range obtained in present the study is in agreement with the results of other studies: pH 3 in Al-Homaidan *et al.* (2016), pH 5 in Gong *et al.* (2005) and Kőnig-Péter *et al.* (2015) studies.

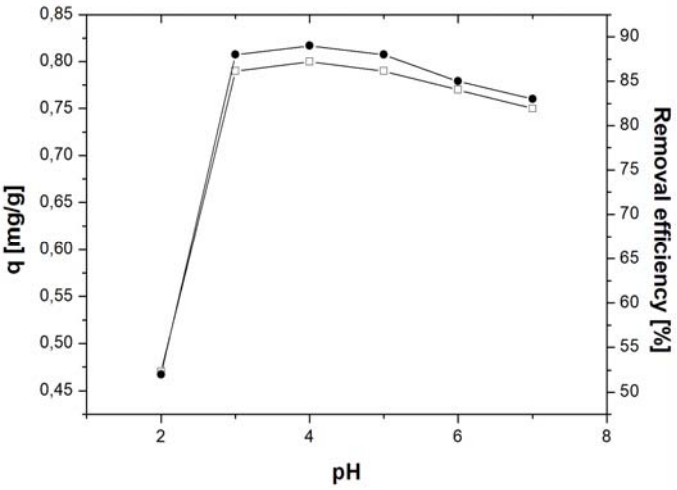

Fig. 1. Removal of lead ions at different initial pH (T 20 °C; C_0 10 mg/L; sorbent dosage 0.5 g; adsorption time 1 h).

Effect of sorbent dosage on biosorption

The effect of spirulina biomass dosage on the biosorption of lead ions was studied using different quantities of biomass (0.1, 0.2, 0.3, 0.4, 0.5, 0.7 and 1.0 g) and maintaining constant all other experimental parameters. As it is shown in Fig. 2, while the biosorbent doses increase from 0.1 to 1 g the percentages of lead adsorbed increased from 54% to 88%. At increase of biosorbents amount, more binding sites are available for metal capture and thus the removal efficiency ramps up.

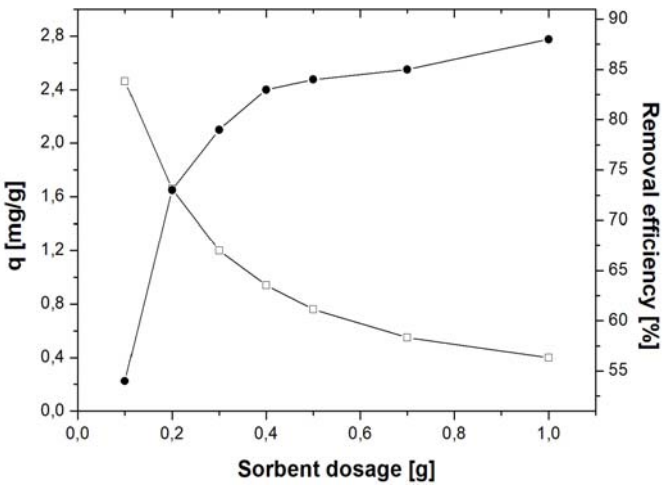

Fig. 2. Effect of biosorbent dose on biosorption capacity and removal efficiency of lead ions by *S. platensis* biomass (T 20 ˚C; C_0 10 mg/L; pH 5; adsorption time 1 h).

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However, the amount of lead adsorbed decreased from 2.46 to 0.4 mg/g. This is due to the fact that at higher adsorbent doses the solution's ion concentration drops to a lower value and the system reaches equilibrium at lower values of 'q' indicating the adsorption sites remain unsaturated (Amarasinghe and Williams 2007).

In Al-Homaidan *et al.* (2016) study, who studied the effect of different biomass dosages (0.25, 0.5, 0.7 5, 1.0, 1.5, and 2.0 g/L) of spirulina biomass on biosoption process the optimum biomass dosage was selected as 2 g. Data obtained by Kőnig-Péter *et al.* (2015) showed that at sorbent dosage of 1 g the lead removal efficiency by cyanobacteria was higher than 80 % and with increasing the biosorbent concentration to 2.0 g the adsorbed amount increased not more than 10 %.

Thermodynamic study

Industrial effluents often are discharged at relatively high temperature, thus temperature is an important parameter affecting the sorbent biosorption capacity. In the present study the solution temperature has varied in the range of 20 – 50°C. A maximum adsorption capacity was obtained at the temperature of 30°C.

The thermodynamic constants namely, free energy change (ΔG°), enthalpy change (ΔH°) and entropy change (ΔS°) were calculated to evaluate the thermodynamic feasibility of the process and to confirm the nature of the adsorption process. The values of enthalpy (ΔH°) and entropy (ΔS°) were evaluated from the slope and intercept of ln K_d *vs.* 1/T plots (Fig. 3). where (ΔH◦), (ΔS◦), T (in Kelvin) and R are the enthalpy, the entropy, the temperature and the gas constant, respectively.

The distribution coefficient (Kd) was calculated using the following formula:

$$K_d = \frac{(C_i - C_e)V}{mC_e}$$

where C_i and C_e are initial and equilibrium lead concentrations (mg/L), V is the volume of the solution (mL), and m is the mass of the biosorbent (g) (Chen and Gao 2009).

The ΔG◦ was calculated from the equation:

$$\Delta G^0 = \Delta H^0 - T\Delta S^0$$

The values of ΔH°, ΔS° and ΔG° are given in the Table 1.

Table 1. Thermodynamic parameters for lead biosorption on *S. platensis*.

Temperature K	ΔG° kJ/mol	ΔH° kJ/mol	ΔS° J/mol·K
293	-1.53	-0.83	34.8
303	-1.87		
313	-2.2		
323	-2.5		

Negative ΔG° values of lead biosorption by spirulina biomass indicate the spontaneity of the adsorption process. ΔG° was more negative with decreasing temperature, which suggested that a lower temperature makes the adsorption easier (Chen *et al.* 2006). ΔH° value -0.83 kJ/mol revealed

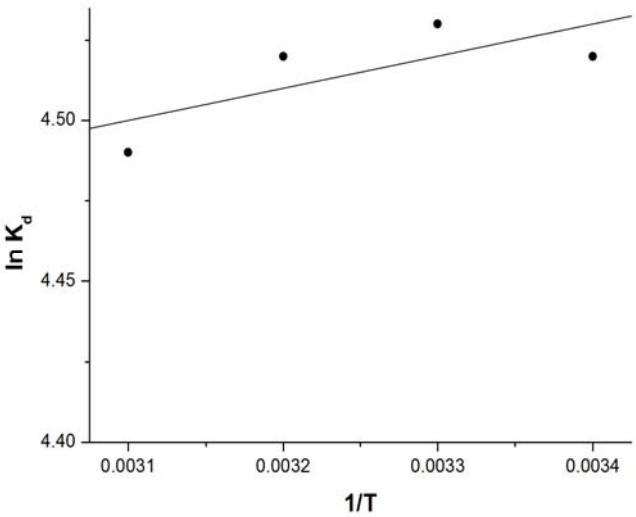

Fig. 3. Dependence of lnK_d *vs.* 1/T.

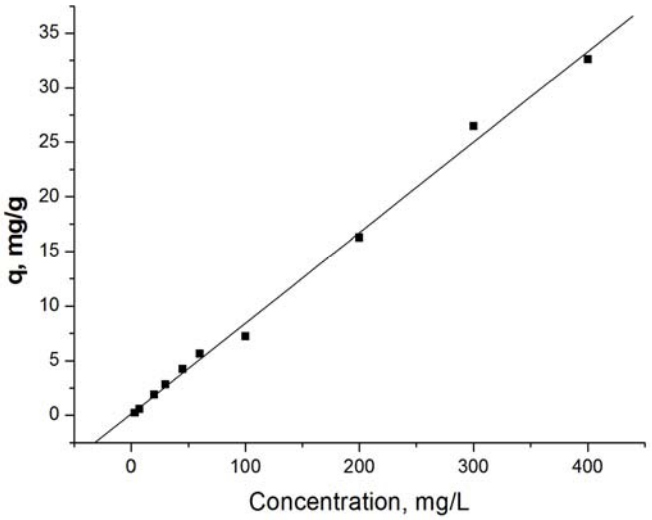

Fig. 4. Freundlich isotherm model.

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that the adsorption process was exothermic in nature, which agrees with the results obtained for the other sorbents (Amini *et al*. 2013; Silva-Medeiros *et al.* 2016). At the same time, a positive value of ΔS° reflects an increased randomness at the solid-solution interface during biosorption and it also indicates that ion replacement reactions occurs (Liu and Liu 2008). Therefore, lead biosorption by spirulina biomass can be considered as a physico-chemical adsorption process rather than a pure physical or chemical adsorption.

Biosorption equilibrium modeling

The Freundlich and Langmuir isotherms are commonly used in biosorption studies to predict the adsorption capacity of a biosorbent. The Langmuir model considers sorption by monolayer type and supposes that all the active sites on the sorbent surface have the same affinity by the sorbate (Li *et al.* 2010). This equation of the Langmuir isotherm is expressed as follows:

$$q_e = \frac{q_m b C_e}{1 + b C_e}$$

where q_e (mg/ g) is the amount adsorbed at the equilibrium concentration C_e, q_m (mg/g) is the Langmuir constant representing the maximum monolayer adsorption capacity and b (mol/L) is the Langmuir constant related to energy of adsorption. The main advantage of this model is the possibility to evaluate maximum possible quantity of metal ions adsorbed per gram of adsorbent and constant related to the affinity of binding sites for the metal ions (Miranda *et al.* 2013).

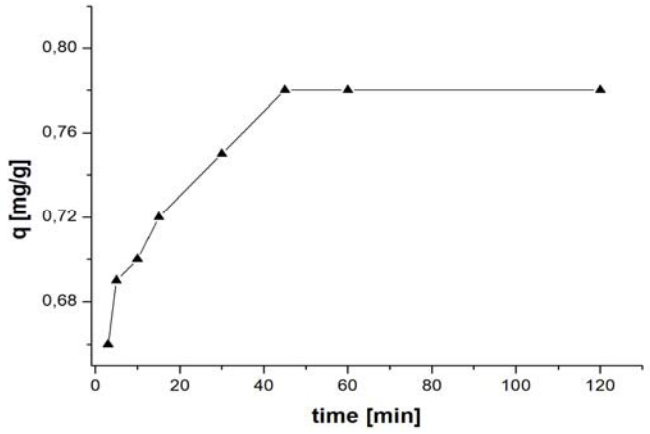

Fig. 5. Effect of contact time on the sorption of lead ions by *S. plat*ensis biomass (T 20 °C; C_0 10 mg/L; pH 5; sorbent dosage 0.5 g).

The Freundlich isotherm is an empirical equation, which assumes a heterogeneous biosorption system with different active sites. The general Freundlich equation is written as follows:

$$q_e = K_F C^{\frac{1}{n}}$$

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

The isotherm parameters have been evaluated by non-linear regression. Obtained data have shown that the adsorption of lead ions by *S. platensis* biomass was unfavorable at the specified conditions for Langmuir model. At the same time experimental data were well described by Freundlich model (Fig. 4, Table 2).

Obtained R^2 value for Freundlich model was higher compared to R^2 value obtained for Langmuir model (data not shown), indicating that the adsorption of lead ions by *S. platensis* biomass was unfavorable at the specified conditions for Langmuir model.

In the Freundlich model, the n values in the range of 1 – 10 indicated favorable adsorption. Applicability of Freundlich model for description of lead biosorption by spirulina species was shown in Al-Homaidan *et al.* (2016) and Gong *et al.* (2005) works.

Biosorption kinetics

As it is shown in Fig. 5 the lead biosorption process by spirulina biomass took place in two stages: the first rapid stage, was achieved in 30 min, and the second equilibrium stage. Lead biosorption capacity reached equilibrium value in

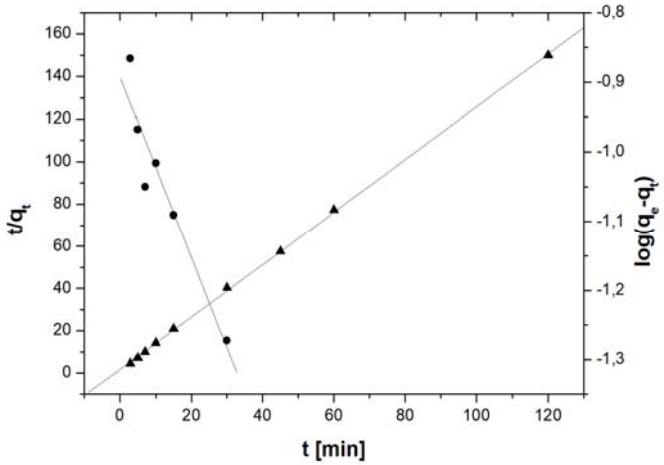

Fig. 6. The pseudo-first- and pseudo second order plots of kinetic study of lead biosorption on *S. platensis*.

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about 60 min. No further increase in the level of bound lead occurred after this time. This is in accordance with the observations of other similar studies (Qaiser *et al.* 2009).

Table 2. Isotherm parameters for the biosorption of lead ions on *S. platensis* biomass.

R^2	0.995
K_f	0.085±0.006
n	1.03

In order to analyze experimental data a pseudo-first order - and pseudo-second order models were used (Chen *et al.* 2011). The pseudo-first-order Lagergren model expressed as:

$$log(q_e - q) = log\, q_e + \frac{K_a}{2.303}t$$

where q and q_e are the adsorbed amounts (mg/g) at time (t) (min) and at equilibrium time, respectively, k_a (min^{-1}) is the rate constant of the first-order biosorption.

The pseudo-second-order kinetics can be expressed:

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

where q and q_e are the adsorbed amounts (µg/g) at time (t) (min) and at equilibrium time, respectively, k_b (g/mg*min) is the rate constant of the second-order biosorption.

The correlation coefficient values obtained from the plots (Fig. 6) suggest that the sorption process fit well the pseudo-first-order kinetics. R^2 for the pseudo second-order kinetic model is greater than 0.99 and substantially higher than that for the pseudo first-order kinetic model. Besides, the q_e values calculated from the pseudo second-order kinetic model agree well with the experimental data values. The reaction rate constant, k and equilibrium biosorption capacity, q_e were determined from the slope and intercept of the straightline plot and are presented in Table 3.

Table 3. Comparison of pseudo-first and pseudo-second order models parameters.

Pseudo-first-order model				
Ce, mg/L	$q_{e\,(exp)}$, mg/g	$q_{e\,(cal)}$, mg/g	k_a, min^{-1}	R^2
10	0.78	0.41	$7.6*10^{-4}$	0.950
Pseudo-second-order				
Ce, mg/L	$q_{e\,(exp)}$, mg/g	$q_{e\,(cal)}$, mg/g	K_b, g/mg.min	R^2
10	0.78	0.8	0.92	0.999

The applicability of pseudo-second order kinetics model suggested that the lead biosorption was based on chemical reaction, involving the exchange of electrons between biosorbent and metal (Qaiser *et al.* 2009). Many researches have also concluded that the second-order kinetics model is more suitable to describe the lead biosorption kinetics (Qaiser *et al.* 2009; Miranda *et al.* 2013).

FTIR analysis

The FTIR analysis was used for the determination of functional groups, which may be involved in the lead biosorption process. In the control FT-IR spectrum (Fig. 7) some intense characteristic bands were obtained for the functional groups presented in proteins and polysaccharides: –OH (1055, 1397.9 cm^{-1}), $–NH_2$, $S\text{-}CH_2$, $NHC(O)_{amid}$ (1235.0), C=O (1535.0), P=O (1625.0), $N\text{-}CH_2$ (2925.0) and NH_2 (3277.0) groups. After lead biosorption the bands of 1055, 1397.9, 1537.5, and 1625.0 cm^{-1} were shifted to 1070.5, 1401.0, 1538.5 and 1627.5 cm^{-1}, respectively. The change in the wave numbers of these bands suggests that OH, C=O and P=O groups can be involved in the biosorption process of lead ions onto spirulina biomass.

Similar results were obtained by other researchers, who studied lead biosorption by cyanobacteria. For example, Gong *et al.* (2005) have shown that amino and hydroxyl groups were involved in the lead biosorption by *Spirulina maxima*. Hydroxyl, carboxyl and amino groups participated in lead biosorption by *Chroococcus multicoloratus* and *Oscillatoria trichoides* (Miranda *et al.* 2013).

Desorption and reuse

Regeneration of biomass in a scale-up is a crucial process parameter that influences the operating

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cost. The desorption analysis of *S. platensis* biomass for the lead ions removal was performed using four different eluent solutions (0.1 M). Biosorption efficiency remained almost constant when CH_3COOH or NaOH were used. However the desorption efficiency was not more than 20%. High removal efficiency can be explained by increase of the concentration of carboxyl and hydroxyl group under interaction of biomass with abovementioned chemicals. FTIR data shows that

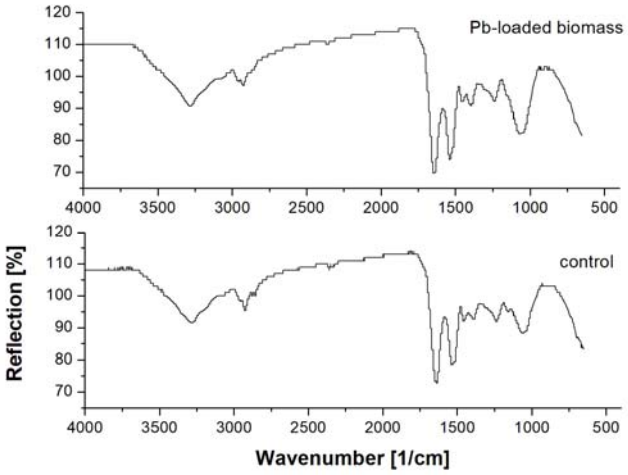

Fig. 7. FT-IR spectra of *S. platensis* biomass: control and Pb-loaded.

these groups are involved in lead biosorption. The applied mineral acids showed good potential as desorption agent for lead with 65 – 67% of recovery in the first cycle (Fig. 8).

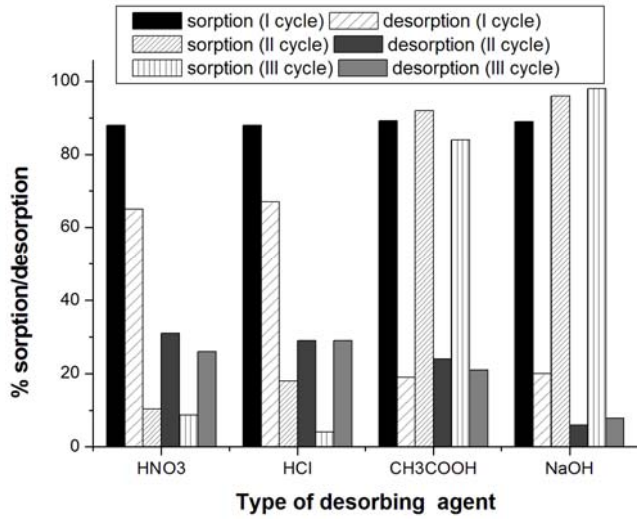

Fig. 8. Biosorption-desorption cycles.

This situation is likely to occur via the exchange of protons with bound lead on cyanobacteria surface. During the next two cycles the desorbing efficiency of biomass treated with mineral acids was drastically reduced, where the percentage removal dropped to 25 – 29% while percentage recovery was 4 – 18% only. The decrease in the biomass sorption/desorption capacity can be explained by structural damage of biosorbent and blockage of binding sites by metal complex. Application of 1.0 M HNO_3 for desorption of Cr(III), Cd(II) and Cu(II) sorbed by cyanobacteria *Spirulina* sp. reduced metals desorption by 50% (Chojnacka *et al.* 2005).

Conclusions

The potential of spirulina biomass application for lead removal in aqueous solutions was demonstrated in the present work. The optimum operating conditions for lead adsorption was found to be 0.5 g biomass, at pH 5, for 30 min. Biosorption equilibrium data fitted very well to both the Langmuir and Freundlich models. Analysis of the data showed that the process involves second-order kinetics. The biosorption of lead by *Spirulina* biomass is an exothermic and spontaneous process. The functional groups of OH, C=O and P=O were mainly involved in lead ions binding by spirulina biomass. The presence of iron ions in the solution affected lead biosorption by spirulina biomass. Mineral acids showed good desorption efficiency in the first cycle, after that it was significantly reduced. Thus it is necessary in further study to find more effective desorbing agent. *S. platensis* can be applied for lead removal from industrial effluents or wastewater post-treatment.

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