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Pb(II) REMOVAL FROM AQUEOUS SOLUTIONS BY USING WASTES OF *PINUS STROBUS* BARK

BY

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Abstract. The influence of significant operational parameters on the Pb(II) sorption process by Romanian *Pinus strobus* has been studied in batch conditions. The uptake of Pb(II) from solutions with different initial concentrations of Pb(II) increased progressively with increasing initial pH from 2 to 5-5.5. The percent removal of Pb(II) varied from 84% to 98% for an increase of bark waste dose to 4 at 60 g/ L. The sorption isotherm studies clearly indicated that the sorptive behavior of Pb(II) ions on Romanian *Pinus strobus* bark under study satisfies not only the Langmuir assumptions, but also the Freundlich assumption. The calculated values for the isothermal thermodynamic parameters show that Pb(II) ions retention by pine bark is a spontaneous process of endothermic and chemical nature. The sorption kinetics for Pb(II) ions removal from aqueous solutions was very well described by pseudo-second order model.

Keywords: *Pinus strobus*; bark; sorption; waste; lead.

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1. Introduction

The need for cheaper and greener alternatives to polymer sorbents has resulted in the search for unconventional materials that may be useful in reducing the levels or accumulation of heavy metals in the environment. A vast array of natural materials and waste products from another production like agricultural, industrial and food production in their raw form or after some physical or chemical modification has been explored as low – cost sorbents of heavy metal ions (Abas *et al.*, 2013; Abdoli *et al.*, 2014; Ahmaruzaman, 2011; Gupta *et al.*, 2015; Dhir, 2014; Nilforoushan *et al.*, 2015). Tree bark, significant wood by-product generated by timber harvesting and timber processing, is among the widely available and low cost sorbents for metal sorption in aqueous environments (Sen *et al.*, 2015). Thus, many studies have been shown that bark of different *Pinus* species (*Pinus brutia*, *Pinus densiflora*, *Pinus pinaster*, *Pinus ponderosa*, *Pinus sylvestris*, *Pinus thunbergii*) has great potential for the removal of toxic metals and organic pollutants and consequently, appear as excellent alternatives to ion exchange resins and activated carbon for industrial applications (Table 1).

Table 1
Research on Pine Bark as Sorbent for Pollutant Removal

Species	Targeted pollutants	Type of study	Reference
<i>Pinus brutia</i>	Pb(II) Cr(VI)	Equilibrium, kinetic and thermodynamic studies; reusability; desorption	(Gundogdu <i>et al.</i> , 2009; Ozdes <i>et al.</i> , 2014)
<i>Pinus densiflora</i>	Cd(II);Cu(II);Zn(II)	Equilibrium studies	(Seki <i>et al.</i> , 1997)
<i>Pinus pinaster</i>	Cd(II); Cr(III); Pb(II) 17 β - estradiol Phenol	Equilibrium and kinetic studies Equilibrium studies Fixed – bed study	(Kumar, 2006) (Braga <i>et al.</i> , 2011) (Vazquez <i>et al.</i> , 2006)
<i>Pinus ponderosa</i>	Cd(II);Cu(II);Zn(II) Ni(II)	Equilibrium, kinetic and dynamic studies	(Oh <i>et al.</i> , 2007)
<i>Pinus sylvestris</i>	Cu(II), Zn(II), Cd(II) 2,4,6 - trinitrotoluene	Equilibrium studies	(Amălinei <i>et al.</i> , 2012; Tofan <i>et al.</i> , 2012) (Nehrenheim <i>et al.</i> , 2011)
<i>Pinus thunbergii</i>	Cd(II);Cu(II);Zn(II)	Equilibrium studies	(Seki <i>et al.</i> , 1997)

Pinus strobus L. (eastern white pine) is a coniferous species native to eastern North America. Owing to its remarkable growth performance, *Pinus*

strobis L. was introduced to Romania in 1861, where has a significant economic importance in Romanian forestry (Blada *et al.*, 2013). *Pinus strobus* L. is a species prized for high production of timber, high quality woods rich in resin, extensively used in the matches and furniture industry, packaging, stationery paste etc. As a result of these uses, large amounts of pine bark wastes are generated, which are valued only by combustion. Through this study we have tried to propose another way to pine bark wastes exploitation and turn them into a potential resource for wastewater treatment in Romania. In this context, we have investigated the sorption removal of Pb(II) ions from simulated aqueous solutions by *Pinus strobus* bark wastes. The choice of Pb(II) as target ion is based on the fact that lead toxicity has been recognized as a major public and environmental health risk. In order to emphasize the sorption characteristics of *Pinus strobus* bark and to establish optimum conditions for Pb(II) uptake, the effects of initial pH, dose of bark, initial cation concentration, temperature and contact time in the Pb(II)- *Pinus strobus* bark batch sorption system have been studied.

2. Materials and Methods

2.1. Plant Material

Patches of *Pinus strobus* bark were collected in the Calimani Mountains (Romania). A full-grown tree was randomly selected for collection. The species was identified and authenticated by specialists from Botanical Garden, Iași, Romania. The bark was shade-dried at room temperature for two weeks and powdered in a knife mill. A voucher sample was deposited in the Department of Pharmacognosy, Faculty of Pharmacy, “Grigore T. Popa” University of Medicine and Pharmacy, Iași, Romania. Before use, the natural material was washed with deionized water several times and then dried at 40°C for 24 h.

2.2. Stock Solution

Analytical grade, $\text{Pb}(\text{NO}_3)_2$ **Sigma-Aldrich** of required quantity was dissolved in double distilled water to prepare stock solution. In order to study the effect of medium acidity on the sorption process, a solution of H_2SO_4 with concentration of 10^{-2} mol/L has been used (340–A /SET 1 pH-meter). Stock solution was diluted with double distilled water to obtain solutions of various concentrations.

2.3. Sorption Procedure

Batch sorption experiments for Pb(II) retention onto *Pinus strobus* bark were performed according to the procedure presented in Fig. 1.

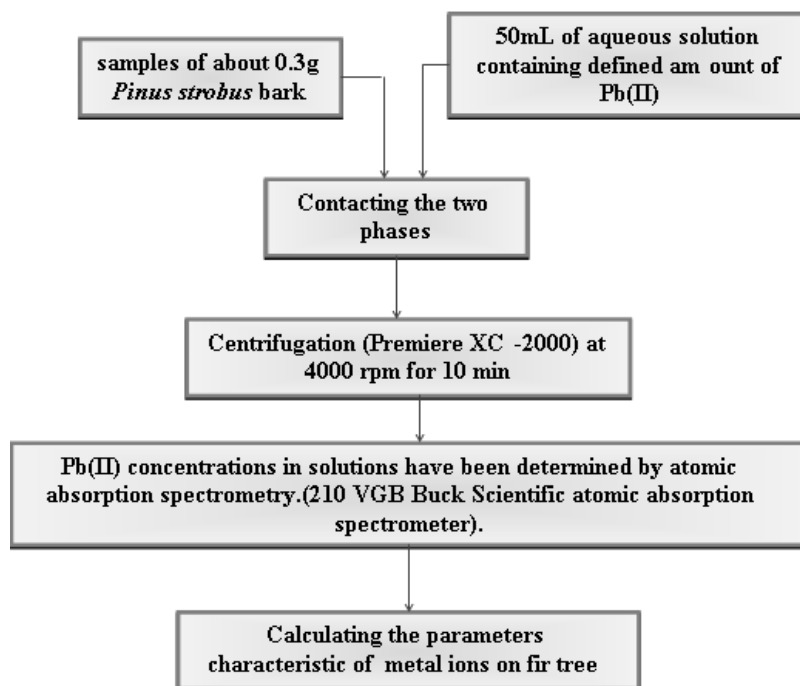


Fig. 1 – Sorption procedure.

The parameters characteristic to the metal ions sorption by pine bark were calculated with the following equations:

$$\text{Retention percentage, } R, [\%] \quad R = [(C_0 - C)/C_0] \cdot 100 \quad (1)$$

$$\text{Retained amount of metal ion, } q, [\text{mg/g}] \quad q = [(C_0 - C)/G] \cdot V \quad (2)$$

where: C_0 – initial concentration of metal ion, [mg/L]; C – cation concentration after sorption, [mg/L]; V – volume of solution, [L]; G – weight of pine bark, [g].

3. Results and Discussion

3.1. Effect of Initial pH

Metal sorption is critically linked with the aqueous phase pH, which governs the speciation of metals and also the dissociation of active functional sites on sorbents (Vankar *et al.*, 2012). This major dependence was investigated in solutions with initial pH in the range of 2 to 7 where the metal under study exists in its double positively charged ionic form (Pb^{2+}) and its precipitation as

metal hydroxide is avoided. The experiments were performed for two initial concentrations of Pb(II) and the results are shown in Fig. 2.

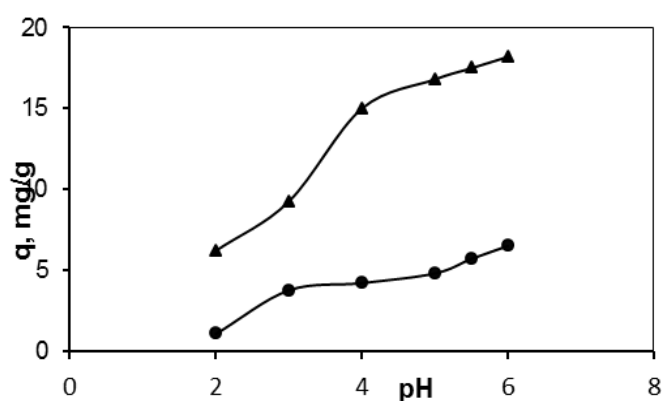


Fig. 2 – The influence of initial pH on the Pb(II) sorption by *Pinus strobus* bark ($C_0 = 60.4$ mg/L(●), $C_0 = 240$ mg/L(▲)).

It can be seen in Fig. 2 that in both cases the sorption of Pb(II) by *Pinus strobus* bark increased with increasing the initial pH. Thus, the uptake of Pb(II) from solution with initial concentration of 60.4 mg Pb(II)/L increased progressively from 1.05 mg/g at pH 2 to 8.8 mg/g at pH 3 and 6.5 mg/g at pH 5-5.5. The respective values for the Pb(II) initial concentration of 240 mg/L were 6.2 mg/g at pH 2 and 18.2 mg/g at pH 5-5.5. This pH - behavior is due to the variation of the charge of *Pinus strobus* bark as function of pH (Seki *et al.*, 1997; Amălinei *et al.*, 2012). The explanation for this increasing trend may be summarized as follows:

	← strong acidic solutions increasing initial pH →	
Charge of bark surface	positive ↓	negative ↓
Pb ²⁺ – bark surface electrostatic interactions	repulsion ↓	strong attraction ↓
Pb(II) sorption on bark	small	more favorable

On the basis of this behavior, the initial pH for the sorption of Pb(II) on *Pinus strobus* bark was optimized as 5-5.5.

3.2. Effect of Sorbent Dose

Fig. 3 illustrates the effect of the bark dose on the Pb(II) sorption from solutions with different initial concentrations (C_0) onto *Pinus strobus* bark wastes.

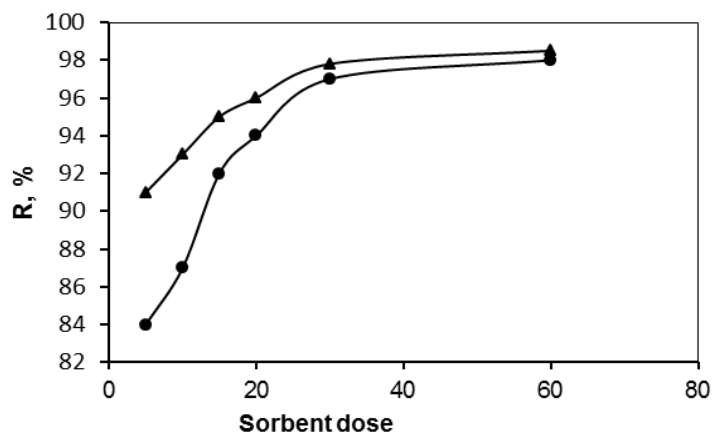


Fig. 3 – Removal percentages of Pb(II) sorption on *Pinus strobus* bark as function of sorbent dose: $C_0 = 60.4 \text{ mg/L}$ (●), $C_0 = 240 \text{ mg/L}$ (▲).

The dependences in Fig. 3 show that the increasing bark dose results in the Pb(II) percentages of retention increase. Thus, for an increase of bark waste dose to 4 at 60 g/L, the percent removal of Pb(II) from solution with an initial concentration of 60.4 mg/L increased from 84% to 98%. At the same time, with an increase in bark dose for 4 to 60 g/L, the efficiency of Pb(II) removal from aqueous solution with initial concentration equal to 240 mg/L increased from 91% to 98.5%. This favorable trend may be due to the increase in sorbent mass → increase in surface area → increasing the number of active sorption sites (Seki *et al.*, 1997).

3.3. Effect of Pb(II) Concentration in Initial Solutions

The influence of metal ion concentration on Pb(II) sorption by *Pinus strobus* bark is presented in Fig. 4. The increase in the initial metal ion concentration produced probably an increase in the driving force of the Pb(II) concentration gradient which leads to the increase of the amount of Pb(II) retained on the tested pine bark (q) (Fig. 4). At the same time, the Pb(II) sorption percentages ($R\%$) decreased with increasing metal ion concentration. This decreasing trend may be attributed to the insufficient binding sites for sorption or to the saturation of the binding sites at higher Pb(II) concentrations (Ozdes *et al.*, 2014).

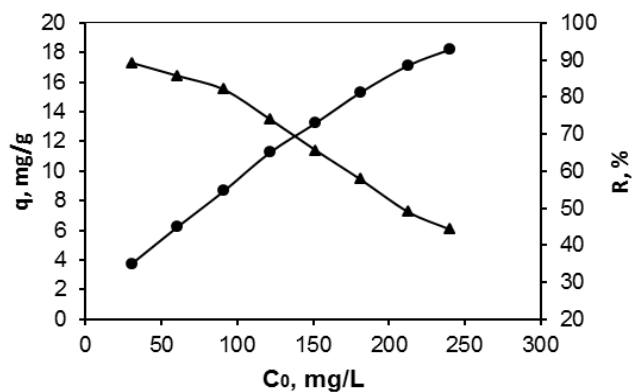


Fig. 4 – Effect of initial concentration (C_0) on Pb(II) sorption by Romanian *Pinus strobus* bark (●) q , mg/g; (▲) R , %.

3.4. Equilibrium Sorption Modeling

One of the physico-chemical aspects with major impact in the assessment of the sorption process as an unitary process is the sorption equilibrium.

The sorption equilibrium is considered established when the concentration of metal ions in the external solution is in dynamic equilibrium with that of the interface.

The functional dependencies between the equilibrium concentrations of Pb (II) in solution and bark phases, at different temperatures, $q = f(C)$, are represented graphically by the Langmuir and Freundlich sorption isotherms in Fig. 5.

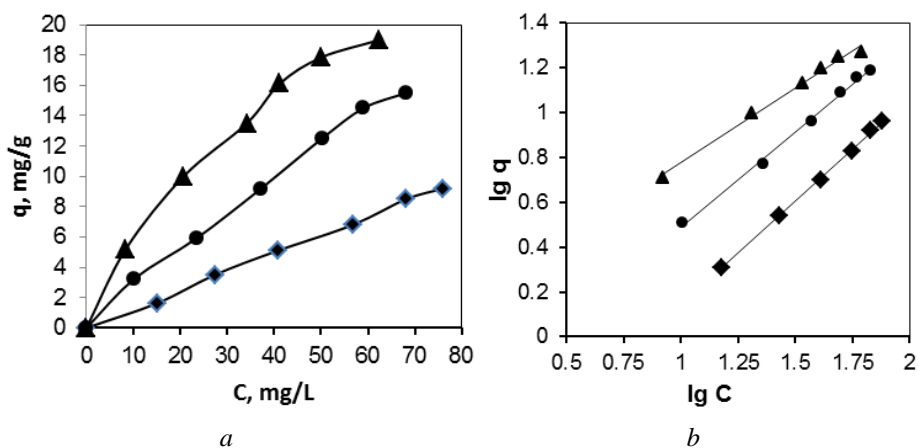


Fig. 5 – Langmuir isotherms (a) and Freundlich isotherms (b) for the sorption of Pb(II) ions on Romanian *Pinus strobus* bark at different temperatures (◆) 277K; (●) 293K; (▲) 333K.

The Langmuir and Freundlich constants characteristic to the Pb(II) - Romanian *Pinus strobus* bark batch sorption system have been obtained from the corresponding linear Langmuir and Freundlich plots and are recorded in Table 2.

Table 2
Isotherm Parameters

Langmuir isotherm (Langmuir, 1916)					
Equation		Quantitative parameters			Isotherm parameters, significance
T [K]		K_L [L/mol]	q_0 [mg/g]	R^2	
$q = K_L \cdot C \cdot q_0 / (1 + K_L \cdot C)$	277	624	8.9	0.9979	K_L – binding energy (relative sorption affinity) q_0 – maximum capacity of sorption
	293	970	10.24	0.9963	
	333	1424	17.56	0.9946	
Freundlich isotherm (Freundlich, 1906)					
$\log q = \log K_F + (1/n)\log C$ (linearised form)		K_F	n	R^2	K_F – sorption capacity n – energy of sorption
	277	1.29	0.951	0.9933	
	293	2.30	1.17	0.9961	
	333	5.26	1.503	0.991	

The values of the correlation coefficients (R^2) in Table 2 point out that the sorptive behavior of Pb(II) ions on Romanian *Pinus strobus* bark under study satisfies not only the Langmuir assumptions (the mechanism of the sorption process as a monolayer sorption on completely homogeneous surfaces where interactions between sorbed molecules are negligible), but also the Freundlich assumption (sorption onto a heterogeneous surface through a multilayer sorption mechanism) (Febrianto *et al.*, 2009, Srivastav *et al.*, 2013). The similar behavior, characteristic for the sorption of Cu(II), Zn(II) and Cd(II) ions on Romanian *Pinus sylvestris* L. bark has been previously reported (Amălinei *et al.*, 2012; Tofan *et al.*, 2012).

It is significant from Table 2 that the values of the K_L Langmuir constant are high enough to reflect the chemical nature of sorption process of Pb(II) ions on *Pinus strobus* bark. On the other hand, the values of the q_0 Langmuir constant (maximum capacity of sorption), obtained for *Pinus strobus* bark are comparable with literature data describing the sorption of Pb(II) by other low cost sorbents (Table 3).

The values of Freundlich constants for the Pb(II)- *Pinus strobus* bark sorption system are recorded in Table 2. The n values are above unity, indicating favorable sorption of the Pb(II) on the pine bark under study.

Table 3
Comparison Between Pb(II) Sorption Capacity of Different Low Cost Sorbents

Low – cost sorbent	Maximum Pb(II) sorption capacity	pH	Reference
Rice husk	0.62	9.0	(Elham <i>et al.</i> , 2010)
<i>Rhizopus arrhizus</i>	2.64	4.5	(Bahadir <i>et al.</i> , 2007)
Waste beer yeast	5.73	5 - 6	(Han <i>et al.</i> , 2006)
Maize husk	7.38	4.0	(Adeogun <i>et al.</i> , 2013)
NaOH–treated fungus <i>Aspergillus niger</i>	8.28	4.0	(Kapoor <i>et al.</i> , 1999)
Untreated olive tree pruning	12.97	5.0	(Ronda <i>et al.</i> , 2013)
Romanian <i>Pinus strobus</i> bark	10.24	4.5 - 5	This study

3.5. Effect of Temperature and Thermodynamic Parameters

The temperature has a favorable effect on the Pb(II) sorption by *Pinus strobus* bark. Both Langmuir and Freundlich constants (Table 2) increase with rinsing temperature, showing that the sorption capacity and the intensity of sorption are enhanced at higher temperatures.

Values of thermodynamics parameters have major significance for practical applications of the sorption process. The changes in free energy, ΔG , enthalpy, ΔH and entropy, ΔS associated with the sorption process of Pb(II) were calculated on the basis of Langmuir constant K_L at different using the following equations (Ozdez *et al.*, 2009):

$$\Delta G = -RT \ln K_L; \quad (3)$$

$$\ln K_L = \text{constant} = - \frac{\Delta H}{RT}; \quad (4)$$

$$\Delta S = \frac{\Delta H - \Delta G}{T} \quad (5)$$

where: R is the gas constant and T is the absolute temperature. ΔH and ΔS values can be obtained from the slope and intercept of Van't Hoff plots of the $\ln K_L$ (from the Langmuir isotherms) versus $1/T$.

The values of thermodynamic parameters as calculated were listed in Table 4. The negative value of the negative free energy change shows that the sorption of Pb(II) on *Pinus strobus* bark is a feasible and spontaneous process. The positive value of ΔH point out the endothermic nature of the process, which is favored by temperature increasing.

Table 4
*The Thermodynamic Parameters of the Sorption
 Proces of Pb(II) on the Pine Bark Under Study*

T [K]	ΔG [kJ/mol]	ΔH [kJ/mol]	ΔS [J/mol·K]
277	-14.80	15.25	0.108
291	-16.75		0.109
333	-20.09		0.106

The positive value of ΔS in Table 4 suggested an increased randomness at the solid/ solution interface during the sorption of Pb(II) ions on the *Pinus strobus* bark.

3.6. Effect of Contact Time and Kinetic Characterization

The kinetics of sorption, describing the Pb(II) sorption rate is an important feature for the efficiency of sorption assessment. The kinetic data for the sorption of Pb(II) by Romanian *Pinus strobus* is shown shown in Fig. 6.

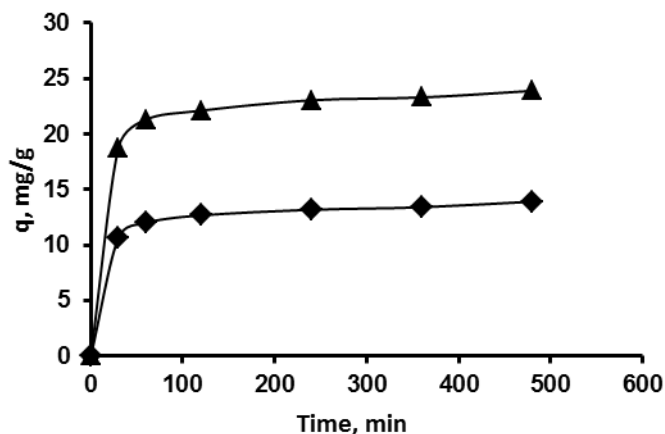


Fig. 6 – Effect of the contact time on the Pb(II) retention by pine bark waste, (◆) $C_0 = 60.4$ mg/L; (▲) $C_0 = 121.48$ mg/L.

The kinetic curves in Fig. 6 show that in the initial stages of the sorption process the amounts of Pb(II) retained on pine bark increased sharply with increasing contact time of phases, attaining values that stayed almost constant.

In order to investigate the mechanism of sorption and potential rate-controlling steps, the pseudo-first-order and pseudo-second-order rate equations were used to test the experimental data:

pseudo-first-order equation:

$$\log (q - q_t) = \log q_e - \frac{k_1}{2.303} \cdot t \quad (\text{Lagergren, 1898}) \quad (6)$$

pseudo-second-order kinetic model:

$$1/q_t = 1/h + (1/q) \cdot t \quad (\text{Ho et al., 1999}) \quad (7)$$

where: q and q_t are the amounts of cation [mg/g] sorbed at equilibrium and at time t , respectively, k_1 is the pseudo-first-order sorption rate constant, [min^{-1}]; k_2 is the rate constant of the pseudo-second-order model and $h = k_2 \cdot q^2$ [mg/g·min] can be regarded as the initial sorption rate constant of the pseudo-second-order sorption, [g/mg·min].

The kinetic parameters obtained from the linearised forms of the pseudo-first-order and pseudo-second-order plots are given in Table 5, together with the corresponding correlation coefficients (R^2).

Table 5
Kinetic Characterization of the Sorption of Pb(II) by Pinus Strobus Bark

Initial concentration C_0 , [mg/L]	q_{exp} [mg/g]	Pseudo-first-order		q [mg/g]	Pseudo-second-order		
		k_1 [min^{-1}]	R^2		k_2 [g/mg·min]	h [mg/g·min]	R^2
60.4	6.25	3.9×10^{-3}	0.9829	7.30	4.75×10^{-3}	0.0946	0.994
121.48	11.25	4.8×10^{-3}	0.966	12.75	6.42×10^{-3}	0.1235	0.9865

Comparing the R^2 values, it is obvious that the sorption of Pb(II) ions on the tested pine bark follows better the pseudo-second-order kinetic model, which is in good agreement with chemisorption being the rate-controlling step (Ho et al., 1999).

4. Conclusions

– The characteristics of Pb(II) sorption from aqueous solution using wastes of *Pinus strobus* bark as a function of pH, sorbent dose, initial Pb(II) concentration, temperature and contact time have been studied under batch conditions.

– Langmuir and Freundlich models were applied to describe the sorption isotherms of Pb(II) by *Pinus strobus* bark.

– The calculated thermodynamic parameters ΔG , ΔH and ΔS , showed that the sorption of Pb(II) onto *Pinus strobus* bark was feasible, spontaneous and endothermic under examined conditions.

– Kinetic evaluation of the experimental data showed that the sorption process followed pseudo-second-order model.

– The obtained results strongly suggest the possibility to use Romanian *Pinus strobus* bark as an effective sorbent for Pb(II) ions removal from aqueous effluents, further research being needed so as to establish the optimum

conditions (technical and economical) for applying such a process to wastewaters.

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ELIMINAREA IONILOR DE PLUMB DIN SOLUȚII
APOASE PRIN UTILIZAREA DEȘEURILOR DE SCOARȚĂ
DE *PINUS STROBUS*

(Rezumat)

S-a studiat, în condiții statice, influența unor parametri operaționali semnificativi asupra procesului de sorbție a Pb(II) pe scoarță de *Pinus strobus* din România. Reținerea Pb(II) din soluții cu diferite concentrații inițiale ale Pb(II) a crescut progresiv la creșterea pH-ului inițial de la 2 la 5-5,5. Gradul de eliminare a Pb(II) a variat de la 84% la 98% pentru o creștere a dozei de scoarță de la 4 la 60 g/L. Comportarea ionilor de Pb(II) în procesul de sorbție pe scoarța de *Pinus strobus* este bine descrisă atât pe baza izotermei Langmuir cât și pe cea a izotermei Freundlich. Valorile calculate ale parametrilor termodinamici arată că reținerea ionilor de Pb(II) pe scoarță de pin este un proces spontan de natură endotermică și chimică. Cinetica sorbției pentru eliminarea Pb(II) din soluții apoase a fost foarte bine descrisă prin intermediul modelului de ordin pseudo-doi.