



Rapid and efficient removal of Pb(II) ions from aqueous media using low quality rapeseeds biomass as biosorbent

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ARTICLE INFO

Article history:

Received 5 August 2017

Received in revised form 21 August 2017

Accepted 21 August 2017

Keywords:

Biosorption

Pb(II) ions

Rapeseeds biomass

Aqueous media

Process modeling

ABSTRACT

In this study, the utilization of low quality rapeseeds biomass as biosorbent for the removal of Pb(II) ions from aqueous media was examined as a function of the most important experimental parameters (initial solution pH, biosorbent dose, contact time, initial Pb(II) concentration and temperature). The experimental results have shown that the most efficient Pb(II) biosorption is obtained at initial solution pH of 6.5, 8.0 g/L biosorbent dose and room temperature (25 ± 2 °C), when more than 93% of Pb(II) ions are retained after 3 h of contact time. The isotherm and kinetics modeling have indicated that the biosorption process of Pb(II) ions on rapeseeds biomass is well described by the Langmuir isotherm model and by the pseudo-second order kinetics model. The value of maximum biosorption capacity, calculated from Langmuir isotherm model (37.3134 mg/g), proves the efficiency of biosorption process, while the high value of pseudo-second rate constant ($k_2 = 0.0701$ g/mg min) indicates the fast rate of Pb(II) removal under these conditions. On the basis of these results, the removal of Pb(II) ions by biosorption on rapeseeds biomass can be considered a rapid and efficient process, which could be successfully used in the industrial wastewater treatment.

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1. Introduction

Environmental pollution with heavy metals is one of the most serious issue in many industrialized countries [1, 2], due mainly to the ecological and human health problems generated by this. In most of cases, the industrial wastewaters are considered the main source of the environment pollution with heavy metals, and from this reason serious efforts are made in order to found the most adequate treatments for reduce the heavy metals content, before the wastewater discharge [3].

Different methods have been tested for the removal of heavy metals from industrial wastewater, such as chemical precipitation, ion exchange, membrane filtration, osmosis, coagulation, electrochemical techniques, etc. [4-7]. Unfortunately, most of these are expensive, require high energy and/or chemical reagents consumption, or generate large amounts of sludge, which must be also treated to not become, in turn, a pollution source for

environment. In comparison with other heavy metals removal methods, the biosorption has attracted great attention in the recent years, because is considered a method with high efficiency, which can be easy controlled during of operating, and involve relatively reduced costs [8, 9]. But, all these advantages become important only if the biosorbent used for the heavy metal removal fulfill the following requests: (i) is easy to obtain, (ii) has high internal surface area, (iii) has high mechanical and chemical resistance, (iv) has on its surface numerous and various functional groups that can interact with heavy metal ions from aqueous solution, and (v) easy to be regenerated or separated from the adsorbate ions [10, 11].

A large number of natural materials, by-products, or industrial and agricultural wastes (such as algae, yeasts, bacteria, fungi, plants leaves and shells, grain straws, etc.) have been used for the removal of different heavy metals in various experimental conditions [10-14]. The general conclusion of these studies is that the selection of a particular biomass as biosorbent for the heavy metal removal is mainly dictated by two major considerations, namely: the

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biosorptive performances of selected biomass, and its availability and preparation cost. These two considerations have been the starting point for the selection, in this study, of rapeseeds biomass as biosorbent for Pb(II) ions removal from aqueous solution.

The rape is a common oleaginous plant, which is cultivated on large surface areas in many regions of the world [15]. The harvested rapeseeds are mainly used in the food industry and for oil extraction for production of biodiesel, but these utilizations are possible only of the rapeseeds meet certain quality requirements. If the rapeseeds do not meet quality requirements (due to unsuitable weather or inadequate cultivation technology), they are milled and used for the animal feed [15, 16]. In order to increase the valorization possibilities, the low quality rapeseeds can be also used as biosorbent for the removal of heavy metal ions from aqueous solution.

In this study, the low quality rapeseeds biomass has been tested as biosorbent for the removal of Pb(II) ions from aqueous media. The lead(II) ions was selected as pollutants for this study both due to their numerous applications in industrial activities (such as storage batteries, paint and pigments, alloys manufacturing, etc.), and due to the great hazard to environment and human health [17]. The experiments were performed in batch systems, as a function of initial solution pH, biosorbent dose, contact time, initial Pb(II) concentration and temperature, in order to found the optimal biosorption conditions. The obtained results were modeled using two isotherm models (Langmuir and Freundlich) and two kinetics equations (pseudo-first order and pseudo-second order models) in order to highlight the efficiency and the rate controlling step of biosorption process.

2. Experimental

2.1. Materials

All the chemical reagents were of analytical degree and were used without further purifications. Stock solution of 1000 mg Pb(II)/L was obtained by dissolving of certain amount of lead nitrate (purchased from Chemical Company SRL, Romania) in distilled water. All the working solutions were prepared by dilution of an exact measured volume of stock solution with distilled water. 0.1 N HNO₃ (Reactivul Bucharest, Romania) solution was used for the initial pH adjustment of working solutions at required value.

The rapeseeds used for biomass preparation were purchased from a local farm (Iași, Romania). Before utilization, the seeds were washed several times with distilled water to remove foreign materials, dried in air at room temperature for 4-5 days, crushed and sieved. The fractions with the grain-size lower than 1.0 mm was kept for the biosorption experiments, and stored in desiccators.

2.2. Methods

All the experiments were performed in batch systems, at mixing speed of 500 rpm, for different values of initial solution pH, biosorbent dose, contact time, initial Pb(II) ions concentrations and temperature. The influence of initial solution pH was examined by contacting 0.2 g of rapeseed biomass with 25 mL of Pb(II) solution (41.43 mg/L) for 24 h and adjusting the values of initial solution pH in the interval between 1.0 and 6.5. In case of biosorbent dose, the

same volume of 25 mL of Pb(II) solution with the same concentration (41.43 mg/L) and initial pH of 6.5, was mixed with different amount of biosorbent (0.1 - 0.5 g), for 24 h of contact time. The effect of contact time was studied using a constant dose of biosorbent (0.2 g), 25 mL of Pb(II) solution (41.43 mg/L) and initial pH of 6.5, but changing the time of components contacting between 5 and 180 min. The influence of initial Pb(II) ions concentration on the biosorption capacity of rapeseed biomass was investigated in the 20.71–497.23 mg Pb(II)/L concentration range, at initial solution pH of 6.5, 8.0 g/L biosorbent dosage and 3 h of contact time. The influence of temperature was examined using 25 mL of different Pb(II) solutions (with concentration in mentioned range) which were mixed with 0.2 of rapeseeds biomass at pH of 6.5 and 3 h of contact time.

At the end of each series of experiments, the biosorbent was separated from solution by filtration, and Pb(II) ions concentration in filtrate was analyzed spectrophotometrically using 4-(2-pyridilazo)-resorcinol as colour reagent (Digital Spectrophotometer S104D, 1 cm glass cell, $\lambda = 530$ nm, against blank solution) and a prepared calibration graph.

In order to ensure the accuracy, all batch biosorption experiments were performed in duplicate and the average values of two data were used for the calculation of biosorption capacity (q , mg/g) and percent of Pb(II) removal (R , %), according with the relations:

$$q = \frac{(c_0 - c)(V/1000)}{m} \quad (1)$$

$$R \% = \frac{(c_0 - c)}{C_0} 100 \quad (2)$$

where: c_0 is the initial concentration of Pb(II) ions in the solution (mg/L), c is the equilibrium concentration of Pb(II) ions in the solution (mg/L), V is volume of solution (mL), and m is the mass of the biosorbent (g).

3. Results and discussion

3.1. Optimization of experimental parameters

Many studies from literature [18-20] have shown that the biosorption of heavy metals occurs with maximum efficiency only in well-defined experimental conditions. Therefore, before to evaluate the utility of certain biosorption process in the treatment of industrial wastewater, the optimal values of experimental parameters must be established. This can be done through batch experiments, and the most important parameters which should be considered, are initial solution pH, biosorbent dose, contact time, initial concentration of metal ions and temperature.

3.1.1. Influence of initial solution pH

Always, the initial solution pH is considered the most important experimental parameter which significantly influenced the efficiency of certain biosorption process. This because its value influences both the dissociation degree of functional groups from biosorbent surface and the speciation form of metal ions in aqueous solution [21]. Therefore, it is necessary that the variation of initial solution pH to be done in a quite wide range, in order to obtain useful information which will allows the selection of his optimal

value. In this study, the initial solution pH was varied between 1.0 and 6.5, and the obtained results are presented in Fig. 1(a).

As can be observed from Fig. 1(a), the biosorption capacity increases with the increase of initial solution pH, and the maximum values are obtained in the pH range of 5.4–6.5. This variation is mainly determined by the increasing of dissociation degree of superficial functional groups from rapeseeds biomass with the increasing of pH, which will facilitate the electrostatic interactions with positively charged Pb(II) ions. The importance of electrostatic interactions in biosorption mechanism is highlighted by the low values of q and R parameters in strong acid media, where the protons concentration is high, and their presence will inhibit the dissociation of biosorbent functional groups [4, 13].

In order to select an optimal value for the initial solution pH, the pH of solutions obtained after filtration (final pH) was also measured (Fig. 1(b)). The obtained results have indicated that the increase of initial solution pH over 5.4 value does not change too much the pH final values (5.29–5.39). The almost constant values of the final pH (obtained even if the initial solution pH was increased over one unit) explain the close values of the biosorption parameters obtained in this interval, and highlight the ability of the rapeseeds biomass to adjust the pH of aqueous solution during biosorption.

Therefore, analyzing all these aspects, an initial solution pH of 6.5 was considered as optimal and was used in further experiments.

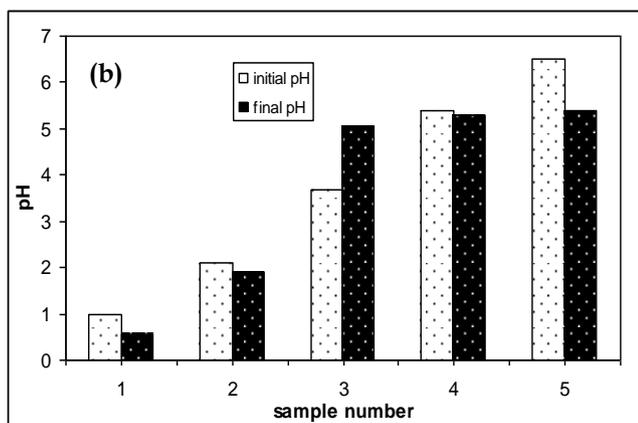
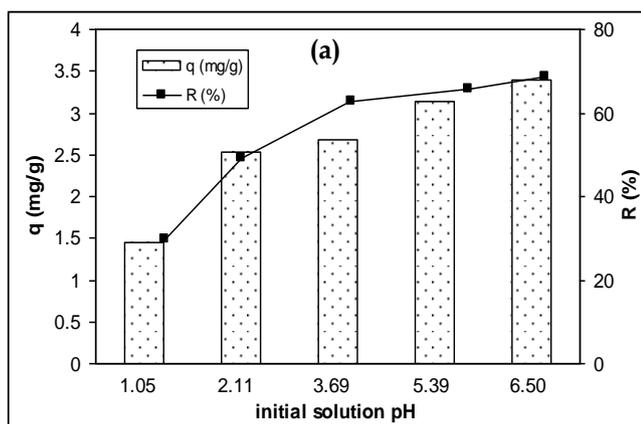


Fig. 1 - Influence of initial solution pH on the biosorption efficiency of Pb(II) ions on rapeseeds biomass.

3.1.2. Influence of biosorbent dose

The effect of biosorbent dose on the Pb(II) ions biosorption capacity of rapeseed biomass is illustrated in Fig. 2. The biosorption capacity of rapeseeds biomass decreases from 7.67 to 1.77 mg/g as biosorbent dose increase from 4 to 20 g/L, while the removal percent values are rising slightly from 81.14 to 89.47 %.

The increasing in the biosorption percentage is obvious due to the increase in the biosorbent quantity, which makes that the number of biosorption sites to be higher. However, the slight increase of R values clearly indicates that for an initial concentration of Pb(II) of 41.43 mg/L, the number of biosorption sites are sufficient even at low values of biosorbent dose.

On the other hand, the decrease of q values is a consequence of the decreasing of the ratio between number of Pb(II) ions (which is constant) and number of available biosorption sites (which increase with the increasing of biosorbent dose). Under these conditions, a biosorbent dose of 8.0 g/L was selected as optimal for the biosorption experiments, both for economic and efficiency considerations.

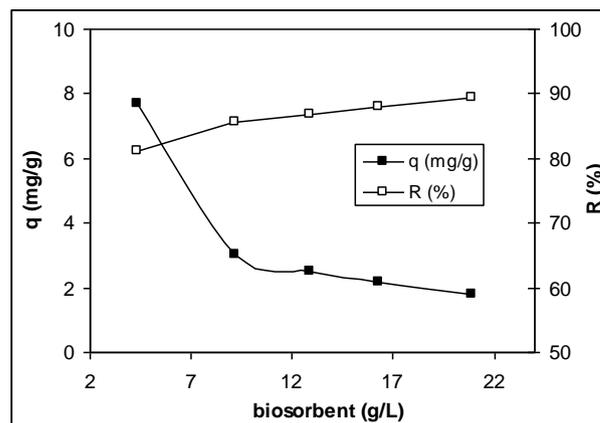


Fig. 2 - Influence of biosorbent dose on the biosorption efficiency of Pb(II) ions on rapeseeds biomass.

3.1.3. Influence of contact time

Fig. 3 represents the variation of biosorption capacity of rapeseeds biomass in function of contact time, at room temperature ($25 \pm 2^\circ\text{C}$), initial solution pH of 6.5 and biosorbent dose of 8.0 g/L. It is quite evident from Fig. 3 that the biosorption of Pb(II) ions ($c_0 = 41.43$ mg/L) on rapeseeds biomass is a very fast process and the equilibrium is reached in 20 min.

Thus, most of Pb(II) ions are removed in the first 5 min, followed by the gradually increase of biosorption capacity up to 20 min, where the removal percent reaches around 75 %, after that remains almost constant until of 180 min of contact time. On the basis of obtained experimental results, a contact time of 180 min between rapeseeds biomass and Pb(II) ions solution was considered enough for the quantitative removal of Pb(II) ions, and was selected as optimal.

3.1.4. Influence of initial Pb(II) ions concentration

Fig. 4 shows the effect of the initial Pb(II) ions concentration on his biosorption efficiency using rapeseeds biomass as biosorbent, under experimental conditions established as optimal. The

experimental results indicate that the biosorption capacity of rapeseeds biomass gradually increases (from 1.31 to 35.85 mg/g) with the increase of initial Pb(II) ions concentration, on entire studied initial concentration interval (20.72–497.23 mg/L). This variation can be attributed to the fact that the increase of Pb(II) ions concentration provide a higher probability of collision between metal ions and biosorbent surface, and it is a typical behavior reported in many studies from literature [20, 22-24].

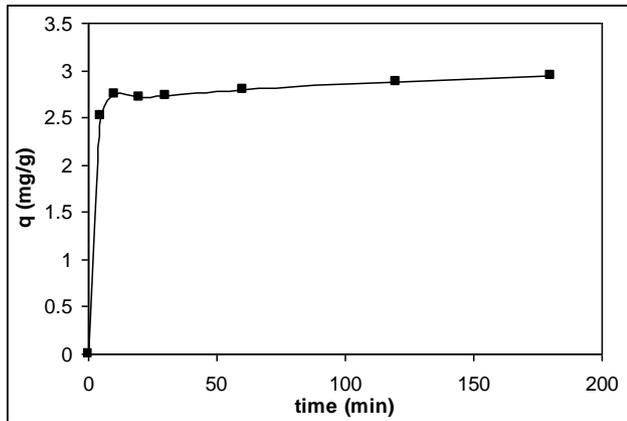


Fig. 3 - Influence of contact time on the biosorption efficiency of Pb(II) ions on rapeseeds biomass.

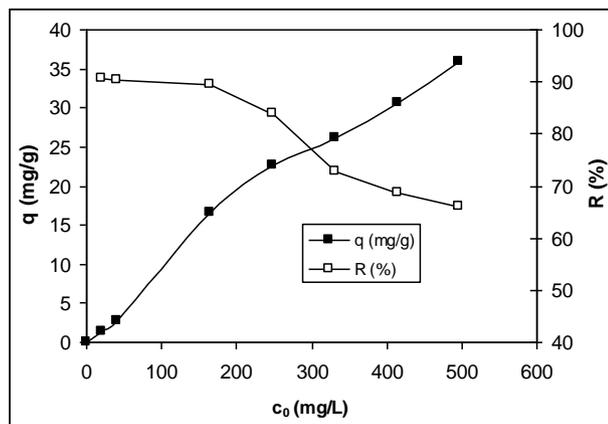


Fig. 4 - Influence of initial metal ions concentration on the biosorption efficiency of Pb(II) on rapeseeds biomass.

On the other hand, the values of removal percent decreases with the increase of initial Pb(II) ions concentration, from 90.74 % ($c_0 = 20.71$ mg Pb(II)/L) to 66.15 % ($c_0 = 497.23$ mg Pb(II)/L). These relatively high values of removal percent, obtained on entire interval of initial Pb(II) ions concentration, suggests that the rapeseeds biomass can be used for the efficient biosorption of Pb(II) ions from aqueous media. Unfortunately, the experimental results have indicated that the quantitative removal of Pb(II) ions through biosorption on rapeseeds biomass is obtained only at relatively low initial metal ions concentrations (below 10 mg Pb(II)/L), when the concentration of Pb(II) ions in effluent solution (obtained after

biosorption) is lower than the maximum permissible limit [25]. Therefore, the industrial effluents which have a Pb(II) ions content lower than 10 mg/L, can be treated through biosorption using rapeseeds biomass as biosorbent in a single step, while the industrial effluents with a Pb(II) ions content higher than 10 mg/L, required two or more biosorption steps to reduce the metal ions content below the permissible limits.

3.1.5. Influence of temperature

In case of Pb(II) ions biosorption on rapeseeds biomass, the temperature has an insignificant effect. The experimental studies have shown that the rise of temperature from 10 to 55 °C determined a decrease of biosorption capacity with maximum 6 mg/g, at highest initial Pb(II) concentration of 497.23 mg/L (Fig. 5).

Under these conditions, it was considered that the optimal value is the ambient temperature, because this is most easily and cost-effectively achieved.

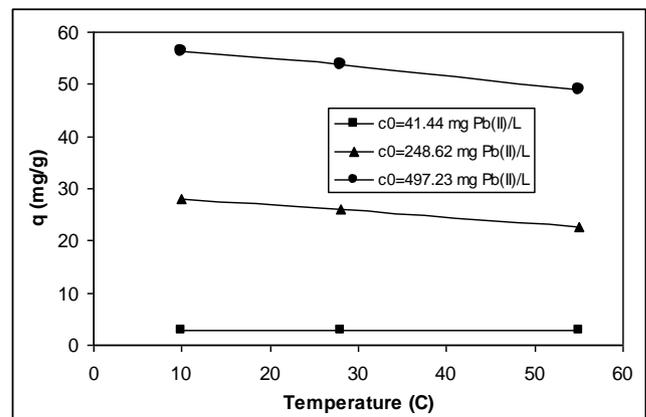


Fig. 5 - Influence of temperature on the biosorption efficiency of Pb(II) on rapeseeds biomass.

3.2. Biosorption equilibrium

The biosorption equilibrium of Pb(II) ions removal on rapeseeds biomass was analyzed using Langmuir and Freundlich isotherm models. The mathematical equations of these two models and the isotherm parameters, calculated from the linear representation of each model, are summarized in Table 1. By comparing the values of regression coefficients (R^2) of the Langmuir and Freundlich isotherm models (Fig. 6) it can be observed that the Langmuir model ($R^2=0.9801$) is more suitable to describe the biosorption process of Pb(II) ions on rapeseeds biomass, than the Freundlich isotherm model ($R^2=0.7821$).

Therefore, the biosorption of Pb(II) ions from aqueous solution occurs until at the formation of monolayer coverage on the outer surface of biosorbent, and demonstrates that the rapeseeds biomass surface is up of homogeneous biosorption patches [20, 23].

According to the Langmuir model, the maximum biosorption capacity of rapeseeds biomass is 37.31 mg/g, and it is comparable to the values obtained at the utilization of other agricultural wastes as biosorbents for the removal of Pb(II) ions, in similar experimental conditions (Table 2). This suggests the potential application of this biomass in the treatment processes of industrial wastewater.

Table 1 - Isotherm characterization of Pb(II) biosorption on rapeseeds biomass.

Model	Linear equation	Notations	Parameters values
Langmuir	$\frac{c}{q} = \frac{c}{q_{max}} + \frac{1}{q_{max}K_L}$	q - biosorption capacity at equilibrium q_{max} - maximum biosorption capacity K_L - the Langmuir constant	$R^2 = 0.9801$ $q_{max} = 37.3134 \text{ mg/g}$ $K_L = 0.0442 \text{ L/mg}$
Freundlich	$\log q = \log K_F + \frac{1}{n} \log c$	K_F - Freundlich constant n - heterogeneity factor	$R^2 = 0.7821$ $n = 1.0713$ $K_F = 0.4029 \text{ L/mg}$

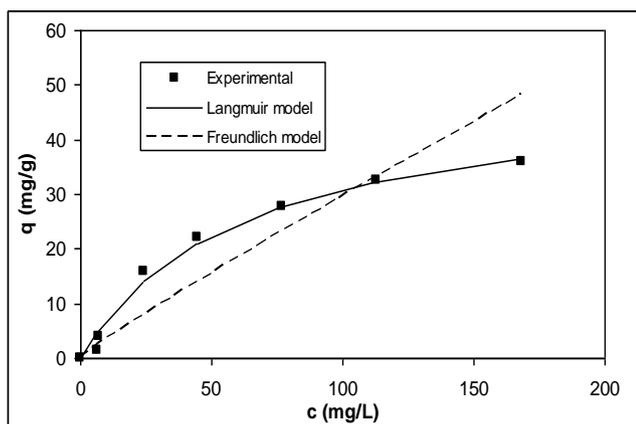


Fig. 6 – Langmuir and Freundlich isotherms representations for Pb(II) ions biosorption on rapeseeds biomass.

Table 2 - Values of maximum biosorption capacity obtained at the biosorption of Pb(II) ions on various agricultural wastes biosorbents.

Biosorbent	pH	q_{max} (mg/g)	Reference
Corncoobs	5.0–5.5	8.29	[26]
Peanut hulls	5.0–5.5	30.04	[26]
Grape bagasse	3.0	42.27	[27]
Sugar beet pulp	5.0–5.5	0.37	[28]
Tea waste	5.0–6.0	48.00	[29]
Mustard biomass	5.5	81.73	[30]
Mustard waste biomass	5.5	73.78	[18]
Rapeseeds biomass	6.5	37.31	This study

Also, the high value of Langmuir constant (K_L , L/mg), which is related to the energy of biosorption process, indicates that between superficial functional groups of rapeseeds biomass and Pb(II) ions from aqueous solution, strong interactions occurs, probably ion-exchange type [4, 11]. But from mechanistic point of view, to understand the nature of biosorbent-metal ions interactions further researches are required in this direction.

Although, the Freundlich model not so adequate to describe the experimental results, the values of n constant close to unit, shows that the biosorption process of Pb(II) ions on rapeseeds biomass is spontaneous even at high concentration of metal ions [9].

3.3. Biosorption kinetics

The kinetics of Pb(II) ions biosorption on rapeseeds biomass was modelled using pseudo-first order and pseudo-second order kinetics models. The linear representations of these two models are illustrated in Fig. 7, while the values of characteristics parameters, calculated from regression equation of each model are summarized in Table 3.

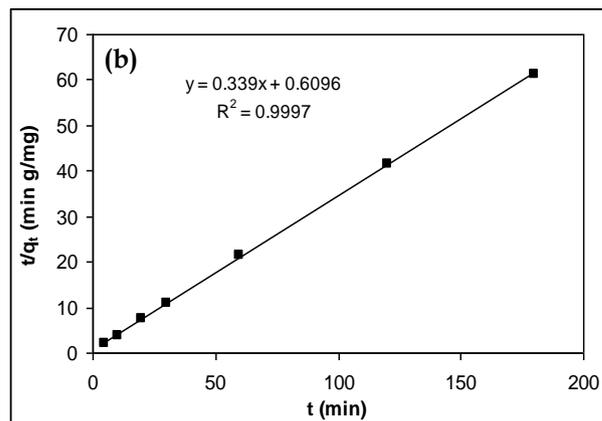
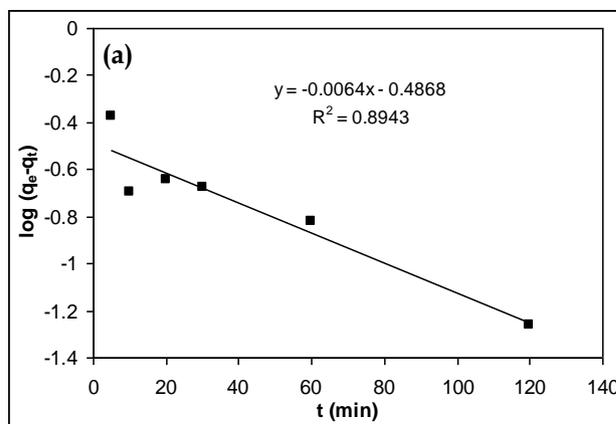


Fig. 7 – Linear representation of pseudo-first order (a) and pseudo-second order (b) kinetics models for the Pb(II) ions biosorption on rapeseeds biomass.

Table 3 - Kinetics characterization of Pb(II) biosorption on rapeseeds biomass.

Model	Linear equation	Notations	Parameters values
Pseudo-first order	$\log(q_e - q_t) = \log q_t - k_1 t$	q_e, q_t - biosorption capacity at equilibrium and at time t k_1 - the pseudo-first order rate constant	$R^2 = 0.8943$ $q_e = 0.3259$ mg/g $k_1 = 0.0064$ 1/min
Pseudo-second order	$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$	q_e, q_t - biosorption capacity at equilibrium and at time t k_2 - the pseudo-second order rate constant	$R^2 = 0.9997$ $q_e = 2.9498$ mg/g $k_2 = 0.0701$ g/mg min

Based on the regression coefficients values (R^2), it can be observed that the pseudo-second order kinetics model is more suitable to describe the biosorption mechanism of Pb(II) ions on rapeseeds biomass, in mentioned experimental conditions. The good correspondence between experimental results and pseudo-second kinetics model is also sustained and by the close values of equilibrium biosorption capacity (q_e , mg/g) obtained experimental (2.9397 mg/g) and calculated on the basis of pseudo-second order kinetics equation (2.9498 mg/g) (Table 3).

According to the pseudo-second order kinetics model assumption, the rate limiting step of the Pb(II) ions biosorption on rapeseeds biomass is the chemical interaction between metal ions from aqueous solution and superficial functional groups of biosorbent. Furthermore, the high value of the rate constant of this model (see Table 3) indicates that in the biosorption process is involved strong interactions between these two partners (probably, ion exchange interactions). Such behavior is frequently reported in literature for the biosorption of various metal ions on different biosorbents of agricultural origin [10, 24], and proves the ability of these biomasses to act as ion exchangers.

Looked at as a whole, the Pb(II) ions biosorption on rapeseeds biomass is controlled by ion-exchange mechanism, in which are involved the superficial functional groups of biosorbent. This makes that the efficiency of biosorbent to depend by the number of available functional sites. When these biosorption sites are occupied (due their interaction with Pb(II) ions), the biosorption process reach the equilibrium, and this is happened very quickly (after 20 min).

4. Conclusion

In this study, the removal of Pb(II) ions by biosorption using rapeseeds biomass obtained from low quality seeds was investigated, in batch systems. The experiments were performed as a function of most important experimental parameters (initial solution pH, biosorbent dose, contact time, initial Pb(II) concentration and temperature), in order to establish the optimal conditions for biosorption. Thus, it was shown that the higher quantity of Pb(II) ions are retained at initial solution pH of 6.5, 8.0 g/L biosorbent dose and 3 h of contact time, when the obtained removal percent is higher than 93 %. The Langmuir and Freundlich models were used to provide the mathematical description of the

equilibrium data, while the pseudo-first order and pseudo-second order models were used for the kinetics modeling. The experimental results have indicated that the biosorption process of Pb(II) ions on rapeseeds biomass is well described by the Langmuir isotherm model and by the pseudo-second order kinetics model. The maximum biosorption capacity (q_{max}) is 37.3134 mg/g and it is comparable to the values obtained at the utilization of other agricultural wastes as biosorbents for the removal of Pb(II) ions, in similar experimental conditions. Also, the high rate constant calculated from pseudo-second order kinetics model indicates that in the biosorption process, the rate controlling step is the chemical interaction (probably ion exchange type) between functional groups from biosorbent surface and Pb(II) ions from aqueous solution, and that the biosorption process is fast. The results presented in this study show that Pb(II) ions by biosorption on rapeseeds biomass, obtained from low quality rapeseeds, is a rapid and efficient process, which could be successfully used in the treatment of industrial wastewater.

Acknowledgements

This paper was elaborated with the support of a grant of the Romanian National Authority for Scientific Research, CNCS - UEFISCDI, project number PN-III-P4-ID-PCE-2016-0500.

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Please cite this article as: T. (Ionel) Arsenie, L. Bulgariu, Rapid and efficient removal of Pb(II) ions from aqueous media using low quality rapeseeds biomass as biosorbent, *Process Eng. J.* 1 (2017) 34-40.