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Dried Leaves of *Bougainvillea glabra* Plant for the Removal of Lead Ions from Aqueous Solution by Adsorption

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> THE PRESENCE of heavy metals in wastewater poses a critical environmental issue and there is a tendency to use natural biosorbents to overcome this problem. In the present study, the dried leaves of *Bougainvillea glabra* (BLB) were chosen as a biosorbent material for lead removal from aqueous solutions. Batch adsorption experiments were performed under various experimental conditions including: contact time, biosorbent weight, initial ions concentration and solution pH. The results showed that lead removal percentage on BLB reached equilibrium after 90 minutes. The maximum percent removal for lead was found as 84.65% at the initial concentrations of 25mg/L. Data were analyzed by the pseudo first and second order kinetic models and the results indicated that the pseudo-second order model best fitted the experimental data ($R^2 > 0.98$). The biosorption of lead by BLB was also successfully described by the intraparticle diffusion mechanism. Equilibrium models were also applied to fit the data and the Langmuir model showed the best fit ($R^2 > 0.99$). Surface characterization of the biosorbent revealed the presence of many active surface groups capable of binding the adsorbate ions. In conclusion, the study revealed that dried leaves of BLB could be suggested as an eco-friendly biosorbent for the removal of lead ions from aqueous solutions.

Keywords: Biosorption, Equilibrium, Kinetics, Lead, Mechanism, Metal ions.

Introduction

The presence of toxic metal ions in the aquatic environment is considered as a major problem especially because of their persistence and nonbiodegradability. Water contamination from heavy metals has gained significant attention due to its negative environmental effects and the prevailing health concerns (Jagaba et al., 2020a). Lead is one of the most persistent toxic heavy metals found in the environment (Ali et al., 2019). Lead can be found in both industrial effluents and sometimes in drinking water. The industrial sources of lead in water include: acid battery, ceramic and glass manufacturing, metal plating and finishing, printing, tanning, and production of lead additives for gasoline (Reyna et al., 2019). The dissolution of lead from lead pipes into tap water has been identified as a source of drinking water contamination (Chang & Lin, 2019). Human, plants and aquatic exposure to organic pollutants along with heavy and organic metal compounds through water and food can result in chronic and sometimes dangerous acute toxicities (Jagaba et al., 2020b). The exposure to lead has a negative impact on human health (Green & Pain, 2019). Lead accumulates in human bodies and causes abnormal calcium metabolism as well as immune disorders(Chang & Lin, 2019). The permissible level for lead in drinking water is 0.05mg/l according to the US Environmental Protection Agency (EPA), thus a very low concentration of lead in water is very toxic (Saleh et al., 2019).

Several conventional methods have been proposed for wastewater treatment, such methods include chemical precipitation, electrochemical reduction, ion exchange, reverse osmosis,

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coagulation-flocculation, sedimentation, filtration, membrane and biological processes (Jagaba et al., 2018; Li et al., 2010). The major disadvantage of these methods is their high cost. In the recent years, there have been great efforts made by scientists in order to find low cost as well as effective alternative methods. In this respect, adsorption has been found to be superior to other techniques for water treatment in terms of initial cost, flexibility and simplicity of design, ease of operation, insensitivity to toxic pollutants and it does not produce harmful substances like most processes (Mohamed et al., 2019).

Biosorption is very suitable for heavy metals removal from aqueous solutions because of biosorbents availability, cost-effectiveness, ease of processing and high rate of natural affinity to metal ions (Jagaba e al., 2020a). Biosorption is a metabolism-independent uptake of metals by non-living biomass. It is a quick process based on physico-chemical interactions between metal ions and functional groups of the biosorbent cell wall (Flouty & Estephane, 2012). Several authors have reported successful removal of lead ions from solution by different biomass materials including: Typha domingensis leaf powder (Abdel-Ghani et al., 2009), Mansonia wood sawdust (Ofomaja, 2010), Ficus carcia leaves (Farhan et al., 2013), Avena fatua biomass (Areco et al., 2013), etc.

During autumn season, tree and plant leaves are usually found fallen all over the roads, thus it is beneficial to collect this type of biomass and use it in environmental friendly ways. In the present work, we aim at using fallen leaves of *Bougainvillea* plant that is widely found in Egypt in home gardens as well as in many road sides as a biosorbent for lead removal from aqueous solution. The parameters affecting the biosorption operation, the equilibrium, kinetics and mechanism models governing the process will be assessed in order to get a profound view about the suggested biosorption process.

Materials and Methods

Biosorbent preparation

The fallen leaves of *Bougainvillea* (Fig. 1) were collected from local gardens in Egypt. The leaves were washed with tap water to remove any dust and then they were further washed with deionized water. The washed leaves were subjected to sun drying. The dried leaves were

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powdered using a grinder, sieved and used as biosorbent without any pretreatment for lead adsorption. The biosorbent was designated as BLB.



Fig. 1. Bougainvillea plant.

Chemicals

Lead standard solution (1000mg/L) was purchased from Merk co. and the working lead solution (25mg/L) was prepared by diluting the standard with deionized water. Analytical grade NaOH and HCl were also obtained from Sigma Co. and were used to prepare 0.1N solution and used for pH adjustment during the experiment.

Batch biosorption experiments

Biosorption experiments were performed at room temperature (25°C) on a shaking water bath using a known amount of BLB with 50mL solution of lead with the desired concentrations in batch mode. To study the effect of contact time, a series of flasks were prepared containing BLB (4g/L and 8g/L) and mixed with lead solution (25mg/L) then shaken for different time intervals (15, 30,60, 90, 120,150 and 180min). The equilibrium time was determined during this experiment. The effect of initial solution pH was investigated at different pH values (2.5-5.5) to determine the optimum pH for biosorption of lead by BLB. The effect of different lead ions concentration on its removal by Bougainvillea biomass was investigated at different lead solution concentrations (5, 10, 25, 50 and 100mg/L). At the end of each experimental set, the solution was separated from the biosorbent by filtration using ashless filter papers and the remaining lead concentration was determined using an inductively coupled plasma (ICP-OES) Perkin Elmer, Optima 2000DV using Argon and Nitrogen gases; the wavelength for lead determination was set as 220.353nm. A schematic diagram representing the experimental process is given in Fig. 2.



Fig. 2. Schematic representation of the experiment.

Calculations

The amount of lead ions adsorbed at equilibrium (biosorption capacity), q_e (mg/g), was calculated by Equation (1) and lead removal percentage (R %) was calculated by Equation (2), where C_i and C_e are the concentrations of lead ions at the initial time and at equilibrium (mg/L), respectively. V is the volume of the solution (L) and W is the mass of adsorbent used (g).

$$\mathbf{qe} = \frac{Ci - Ce}{W} \star V \qquad \text{Eq. (1)}$$

$$R\% = \left[\frac{C_i - C_e}{C_i}\right] * 100 \qquad \text{Eq. (2)}$$

Kinetic and equilibrium modeling

The kinetics modeling of the biosorption process was investigated by applying the pseudo first and second order kinetic models. The mechanism of the biosorption process was also studied using the intra-particle diffusion model.

The pseudo first order equation is given by Equation (3) (Lagergren, 1898):

$$\ln(q_e - q_t) = \ln q_e - K_1 t \qquad \text{Eq.(3)}$$

where q_e (mg. g^{-1}) and q_t (mg g^{-1}) are the amounts of lead ions at equilibrium and at time t, respectively, k_1 (min⁻¹) is the pseudo first order rate constant.

The pseudo second order model is given by Equation (4) (Ho & McKay, 1998).

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$$
 Eq. (4)

where k_2 (g.mg⁻¹.min⁻¹) is the rate constant of the second order equation.

The weber-Morris intra-particle diffusion mechanism (Weber & Morris, 1963) can be expressed by Equation (5)

$$q_t = k_i t^{0.5} + C_{bi}$$
 Eq. (5)

where q_t is the adsorbed quantity of lead, K_i is the intra-particle diffusion parameter, and $C_{b,i}$ is the thickness of the boundary layer at stage (i).

The biosorption of lead by BLB was also studied using the Langmuir (Langmuir, 1918), Freundlich (Freundlich, 1906) and Temkin (Temkin & Pyzhev, 1940) equilibrium models represented by Equations (6), (7) and (8), respectively.

$$q_{e} = q_{max} bC_{e} / (1 + bC_{e}) \qquad \text{Eq. (6)}$$

$$q_e = k_F C_e^{1/n} \qquad \text{Eq.(7)}$$

$$q_e = a_t + b_t ln C_e \qquad Eq.(8)$$

In these equations, q_e and C_e represent the adsorbent capacity at equilibrium (mg/g) and the concentration of lead ions at equilibrium (mg/L), respectively. In the Langmuir equation, q_{max} is considered the maximum sorption capacity related to the total cover of the surface and b is associated to sorption energy. From the Freundlich model, K_F represents the sorption capacity and 1/n is related to the energy distribution of the sorption sites. The Temkin model constants a_t and b_t are related to the maximum binding energy and heat of adsorption.

Biosorbent characterization

Bougainvillea leaves biomass functional groups were characterized using Fourier Transformation Infra Red (FTIR) technique. The FTIR was recorded using Nicolet, AVATAR FTIR-370 Csl over the working range 200-4000/cm. Elemental analysis of *Bougainvillea* leaves biomass was carried out using a Perkin Elmer Model 2400 elemental analyzer, (USA). The percentages of cellulose, hemicellulose and lignin in the biosorbent were determined using an Ankom 220 Fiber Analyzer (Ankom Technology, Fairport, NY, USA).

Results and Discussion

Effect of contact time

The effect of contact time on the removal of Pb (II) by BLB was studied at two different

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biomass loading weights (4 and 8g/L) and the results are shown in Fig. 3. In these experiments, the contact time between the adsorbent and adsorbate solution varied from 15 to 180 minutes. It was noticed that a biosorbent dosage of 8g/L showed higher removal of lead compared to the dosage of 4g/L. It was previously reported that when the biosorbent was increased, removal of metal ions also increased due to the existence of more active adsorption sites on the biosorbent's surface (Kyzas et al., 2014; Lee & Chang, 2011).



Fig. 3. Effect of contact time on Pb(II) removal onto BLB at a lead concentration of 25mg/L, solution pH 4.5 and biomass loading weights (4 and 8g/L).

The results also showed that the removal of lead increased in the first 60min and remained constant starting from 90min. Thus, the equilibrium time for lead removal onto BLB was recorded to be 90min and further experiments were performed at this equilibrium time. The maximum percent removal for lead was found as 84.65% at the initial concentrations of 25mg/L.

During the first stage of biosorption, metal accumulation takes place initially because of the availability of the large surface area. The diffusion of metal molecules to the inner active sites of the biosorbent takes place in the second slower stage. This was confirmed as a decrease in pH was observed at the end of the experiments which might be due to the release of H^+ ions as a result of ion exchange between Pb^{2+} and proton (Surisetty et al., 2013).

Effect of solution pH

It has been generally agreed that the pH of the solution has a critical role in designing an effective biosorption system. The changes in

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the adsorbate pH had significant effects on the surface functional groups of the adsorbent as well as the Pb (II) ions present in the solution, indicating pH is an important parameter in controlling the adsorption (Onwordi et al., 2019). In the present study, the effect of initial solution pH on lead removal by BLB was investigated in a pH range 2.5 to 5.5 since lead (II) ions precipitation appears at higher pH values (Ay et al., 2012), based on preliminary studies and also based on previous literature that showed that the pH for studying lead adsorption is between 2 and 5 (Yu et al., 2001). Above pH 6.0, precipitations become an issue; therefore, pH values above 6.0 were not studied (Mahdi et al., 2018). The results are presented in Fig. 4; it can be noticed that the removal of lead by BLB was very low at highly acidic pH values and increase up to pH 4.5 with almost no further increase after this. Thus, pH 4.5 was considered the optimum pH and further experiments were performed at this pH value. At very low pH values there is a competition for the available biosorbent sites between a high number of hydronium ions present in solution and the lead ions which results in lower lead ions removal. Upon increasing the solution pH, there is an increase in lead ions removal which is likely due to decreasing hydronium ions concentration. Our results are in agreement with the results previously reported for lead removal onto various biosorbents (Gerçel & Gerçel, 2007; El-Ashtoukhy et al., 2008; Tokimoto et al., 2005; Feng et al., 2011a).



Fig. 4. Effect of initial solution pH on Pb(II) removal onto BLB at a lead concentration of 25mg/L, contact time 90min and biomass loading weights 8g/L.

Biosorption kinetics

Different kinds of mechanisms could be involved in controlling biosorption process such as mass transfer, diffusion control, chemical reactions and particle diffusion (Reyna et al., 2019). Thus, in the present study, the pseudo first and second order kinetic models were applied to the experimental data in order to find the model that best describes the biosorption of lead ions onto BLB. The plots of the pseudo first order and pseudo second order models are shown in Figs. 5 and 6, respectively. The kinetic parameters obtained from each model along with the regression coefficient values of the models are summarized in Table 1. It can be noticed that the pseudo-second order model gave best fit to the experimental data based on its high regression coefficient ($R^{2} > 0.98$). From Table 1, the good fitting of the pseudo-second order model is also confirmed by the very close values of q experimental and q calculated from the model. The suitability of the pseudo-second-order kinetic model suggests that the rate-limiting step may be chemical which involves sorption valance forces through sharing or exchange of electrons between heavy metal ions and the adsorbent (Feng et al., 2011b).



Fig. 5. Pseudo-first order kinetics of lead adsorption by BLB.

TABLE 1.	Calculated	kinetic	parameters.
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Fig. 6. Pseudo-second order kinetics of lead adsorption by BLB.

Biosorption mechanism

The biosorption mechanism for lead removal by BLB was studied using the intra-particle diffusion model by plotting (q.) versus (\sqrt{t}) as shown in Fig. 7. The model's regression coefficients and constants are given in Table 2. The plot revealed multilinearity indicating the involvement of more than one mechanism in the biosorption process of lead using BLB. It can be seen from Fig. 7 that two linear parts with high regression coefficients ($R^{2} > 0.96$) exist. The first line represents the external diffusion by macropore and mesopore (El-Naas et al., 2010) and the second one indicates the micropore diffusion by the intra-particle diffusion (Aksu & Kabasakal, 2004). It can be also observed from Figure 7 that the lines do not pass by the origin which signifies that the intra-particle diffusion does not control the whole process of biosorption of the lead ions by BLB and some other mechanisms along with intra-particle diffusion are also involved in the adsorption process (Tan et al., 2009).

	Pseudo- first orde	er	Ps	eudo- second ord	er	
k _{1 (L/min)}	q _{e,} calc. (mg/g)	\mathbf{R}^2	k ₂ (g/mg.min)	q _{e,} calc. (mg/g)	R ²	q _e (exp.) (mg/g)
0.028	2.04	0.900	0.108	1.40	0.989	1.39

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Fig. 7. Intra-particle diffusion mechanism of lead adsorption by BLB.

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K ₁ (mg/g min ^{-0.5})	С _{ь (1)} (mg/g)	R ²	K ₂ (mg/g min ^{-0.5})	С _{ь (2)} (mg/g)	\mathbb{R}^2
0.074	0.613	0.962	0.210	1.365	0.978

Effect of initial lead concentration

The effect of different lead ions concentration on its removal by BLB is presented by Fig. 8. Percentage removal (%) of lead ions has been found to be higher at lower concentration of lead (25mg/L) with a maximum lead removal of 75% and decreased at higher concentrations. This behavior is expected because at low initial concentration there are a lot of unoccupied sites on the biosorbent's surface and these sites become more occupied at higher metal ion concentrations (Ramakul et al., 2012).



Fig. 8. Effect of lead initial concentration on its removal by BLB at contact time 90min, initial solution pH 4.5 and biomass loading weights 8g/L.

Equilibrium modeling

In order to get more information about the biosorption system of lead ions by BLB,

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the Langmuir, Freundlich and Temkin models were used to evaluate the equilibrium data. The Langmuir, Freundlich and Temkin isotherm plots for Pb (II) are given in Figs. 9, 10 and 11, respectively. The calculated results of the Langmuir, Freundlich and Temkin isotherm constants are given in Table 3. It is observed that the Langmuir isotherm model ($R^{2>}$ 0.99) gives best fitting to experimental data of the lead biosorption onto BLB. The applicability of the Langmuir model suggests monolayer adsorption onto the biosorbent surface which contains a finite number of uniform adsorption sites without migration of adsorbed molecules on the surface (Torab-Mostaedi et al., 2013).



Fig. 9. Langmuir plot of lead adsorption by BLB.



Fig. 10. Freundlich plot of lead adsorption by BLB. TABLE 3. Equilibrium models' calculated parameters.



Fig. 11. Temkin plot of lead adsorption by BLB.

	La	ngmuir			Freundlic	ı		Temkin	
	q _{max.} (mg/g)	b (L/mg)	R ²	K _f	n	R ²	a _t	b _t	R ²
Pb 2+	1.450	0.435	0.993	1.874	3.742	0.866	0.276	0.468	0.846

Elemental analysis and fiber fraction of BLB

Table 4 presents the elemental analysis and composition of BLB. The biosorbent was found to be mainly constituting of carbon, oxygen, and hydrogen along with small quantities of other elements. Also, BLB was found to be mainly composing of cellulose and hemicellulose as well as some lignin. Plant cell walls are generally considered as structures built by cellulose molecules, organized in microfibrils and surrounded by hemicellulosic materials (xylans, mannans), lignin and pectin along with small amounts of protein (Demirbas, 2008). It has been demonstrated that lignocellulosic materials have sorption capacities, which are derived from their constituent polymers and structure. The polymers include extractives, cellulose, hemicelluloses, pectin, lignin and protein. These are adsorbents for a wide range of solutes, particularly divalent metal cations (Malik et al., 2017).

Characterization of BLB by Fourier Transformation Infrared (FTIR)

The functional groups present on any sorbent surface play a very important role in its adsorption efficiency. In the present work, the surface functional groups of BLB were identified by FTIR analysis. The FTIR spectra of BLB before and after biosorption are given in Fig. 12. The spectrum shows some characteristic absorption peaks of plant biomass. The peaks at 3434.59 and 3415.31cm⁻¹ correspond to the overlapping of -OH and -NH peaks (Tang et al., 2013). A peak at 2923.55 cm⁻¹ could be assigned to -CH stretching vibrations of -CH₂ and -CH₂ functional groups (Farhan et al., 2013). Another sharp peak at 1621.84cm⁻¹ was also recorded in the spectrum and was attributed to the presence of an amide group (protein) (Tang et al., 2013). The absorption peaks at 1326.78 and 1090cm⁻¹ could be assigned to the C-O stretching vibration of carboxylic acids and alcohols (Feng et al., 2011). The spectrum peak positioned around 778.1 cm⁻¹ may be due to Si-O-Si linkage (Akhtar et al., 2010). It can be thus concluded that the biomass of Bouvingela contains many surface functional groups that makes it suitable for biosorption. After biosorption, several bands that were observed in the spectrum of BLB were shifted and the intensities were changed. The peaks at 3434.59 and 3415.31cm⁻¹ were shifted to 3423.028 and 3405.67cm⁻¹, respectively. Also, the band at 2923.55cm⁻¹ in the BLB spectrum before biosorption showed much lower intensity in the BLB spectrum after biosorption and appeared at 2924.52cm⁻¹. The sharp peaks at 1621.84 and 1326.78cm⁻¹ before adsorption moved to 1608.34 and 1315.21cm⁻¹, respectively with broadness in the peaks. Also the peak around 778.1cm⁻¹ was shifted to 785.85. From FTIR study, the changes in absorption intensity and the shift in wavenumber of functional groups could be due to interaction of metal ions with active sites of biosorbents (Rao et al., 2018).

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Element	Percentage (%)	
С	46.29	
Н	9.10	
0	32.91	
Ν	1.74	
S	1.32	
Cellulose	32.05	
Hemicellulose	29.60	
Lignin	3.90	



Fig. 12. FTIR spectrum of BLB.

Comparison of BLB with other biosorbents

Table 5 gives a comparison of the maximum biosorption capacities obtained by different biosorbents used for lead removal. The maximum biosorption capacity (mg/g) observed for BLB was to some extent comparable with those found in literature. The differences of lead sorption capacities can be attributed to the differences in the properties of biosorbents such as structure, surface area, porosity, and functional groups (Surisetty et al., 2013; Alghamdi et al., 2019).

 TABLE
 5. Comparison of BLB with other biosorbents.

Biosorbent	Maximum capacity (mg/g)	Reference
Tea leaves	2.08	(Ahluwalia & Goyal, 2005)
Typha domingensis leaves	0.65	(Malik et al., 2017)
Bael tree leaf powder	4.06	(Senthil Kumar & Gayathri, 2009)
Tea waste	1.357	(Mondal, 2010)
BLB	1.46	Present study

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Conclusion

The presented work describes the efficiency of Bougainvillea glabra dry leaves as an environmental friendly biosorbent for lead ions removal from aqueous solution. Bougainvillea glabra dry leaves were capable of removing 85% of lead from solution containing 25ppm of lead at a biosorbent dose of 8g/L. It was noted that the removal process increased with contact time and attained saturation in about 90min. The equilibrium data were characterized by the Langmuir, Freundlich and Temkin isotherms. Isotherm equations and correlation coefficient R² showed that Langmuir isotherm fitted the experimental data better than Freundlich and Temkin for lead ions when using Bougainvillea leaves as biosorbent. The kinetics study showed that the biosorption of lead ions was best described by the pseudo-second order kinetic model and the mechanism of adsorption was intraparticle diffusion mechanism. The study suggests that Bougainvillea biomass can be further explored as an efficient biosorbent for the removal of metal ions from aqueous solution since it is a waste

TABLE 4 . Elemental Analysis and fiber fractions of BLB.

material, very cheap, eco-friendly and non-toxic material.

Conflict of interests: The authors declare that they have no conflict of interest.

Authors contribution: Ghadir A. El-Chaghaby: Conceptualization, methodology, validation, investigation, resources, reviewing, editing, approval of submitted manuscript. Sayed Rashad: Methodology, validation, investigation, resources, original draft preparation. Shereen F. Abd El-Kader: Methodology, validation, investigation, resources, revision of manuscript.

Ethical approval: Not applicable.

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إستخدام اوراق نبات الجهنمية المجففة لإزالة الرصاص بالإدمصاص من مياه الصرف

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تواجه معظم دول العالم في الأونة الأخيرة مشكلة بسبب قلة وعجز موارد المياه العذبة والتي يشبهها بعض العلماء بأن الحرب القادمة هي حرب المياه. ولذا يهتم العلماء بدور هم بالبحث عن إيجاد حلول لهذه المشكلة والتي من أهمها توفير مصادر بديلة للمياه. ومن أهم هذه المصادر هي مياه الصرف والتي قد تكون ملوثة بأحد أكثر الملوثات خطورة وهي العناصر الثقيلة. وقد أدى التوسع العمراني والحضاري والصناعي إلى تلوث مصادر المياه المختلفة بالعديد من الملوثات الخطرة مثل العناصر الثقيلة والتي من أهم أمثلتها الرصاص. لذا يهدف هذا البحث إلى استخدام الأوراق المجففة لنبات الجهنمية لإزالة الرصاص من مياه الصرف بخاصية الإدمصاص. ومن أهم النتائج التي تم التوصل إليها في هذا البحث أن 8 جرام/ لتر من أوراق الجهنمية المجففة (BLB) إستطاعت نزع الرصاص من محلول مائي يحتوي على (25ppm) بكفاءة تصل إلى 85%. كما لوحظ أنه بزيادة الوقت تزداد كفاءة أوراق BLB المجففة على إزالة الرصاص حتى حدوث تشبع لهذه الأوراق المجففة عند الدقيقة 90. باستخدام Langmuir, Freundlich and Temkin isotherms تم وصف نتائج توازن الإدمصاص الحيوي لأيونات الرصاص باستخدام الأوراق المجففة ل BLB. وقد أوضحت المعادلات ومعامل الإرتباط أن " Langmuir isotherm" كان مناسب وأفضل من " Temkinو Freundlich ". كما أظهرت نتائج در اسة النماذج الحركية أن النموذج " pseudo-second order" كان الأفضل في وصف إزالة أيونات الرصاص باستخدام الأوراق المجففة لنبات الجهنمية ك "biosorbent" وأن آلية الإدمصاص هي الإنتشار داخل أوراق النبات. وبذلك تستعرض هذه الدر اسة نبات الجهنمية كأحد أهم ال biosorbents التي يمكن إستخدامها في نزع العديد من العناصر الثقيلة وذلك لأنها تعتبر مُخلف زهيد الثمن، صديق للبيئة وغير ضار.