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KINETIC STUDY OF A SINGLE AND BINARY BIOSORPTION OF CADMIUM AND LEAD ONTO THE DEAD AQUATIC PLANT *LEMNA GIBBA*. BIOSORPTION OF HEAVY METALS BY A DEAD AQUATIC PLANT

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Abstract – Cadmium and lead are recognized as toxic heavy metals even at low concentrations. Thus, their removal is required. The present paper deals with the use of a natural low-cost and environmentally friendly material as a bioadsorbent obtained from a dead aquatic plant (Lemna gibba). The biosorption of Cd and Pb individually or in combination was studied under different experimental conditions such as time effect (0-240 min), concentration of metal ion (0.1 and 1 mg/L), adsorbent dose (0.10, 0.25 and 0.50 g) to examine the operational factors impact on heavy metals removal effectiveness. The dead biomass was characterized by FTIR to provide information about the functional groups responsible for biosorption. Inductively coupled plasma atomic emission spectroscopy was employed to perform quantitative measurement of Cd and Pb (ICP-OES). For the kinetic investigation, pseudo-first order and pseudo-second order, models were used.

The experimental results demonstrated that lead and cadmium adsorption onto Lemna gibba powder occurred quickly, with equilibrium being reached in 120 minutes and 30 minutes, respectively. At 0.1, the greatest removal efficiencies were 84.01% of Pb and 93% of Cd. At 1 mg/L, 73.82%, and 88% of Pb and Cd were removed respectively. After 180 minutes, both metals were effectively eliminated (90%) from the binary system that was contaminated with 0.1 mg/L of each metal. At 1 mg/L for each element, Pb was removed 86% after 90 minutes, and Cd clearance was less (54%).

Keywords: biosorption; dead aquatic plant; cadmium, lead, binary mixture, Kinetics

INTRODUCTION

Contamination of the aquatic environment by different pollutants is a serious global problem. The continuous growth in population, the expansion of urbanization and the rapid development of industrialization led to the release of organic matters and heavy metals (Lalevic et al., 2012; Rezania et al., 2015; Hu et al., 2016; Khallaf et al., 2018; Cao et al., 2019)

Among hazardous contaminants, heavy metals are a common environmental threat (Chiban et al., 2016). The pollution of ground and surface waters with heavy metals is a widespread and a serious problem (Benhima et al., 2008). With the expansion of industry, large quantities of heavy metal–contaminated water are being discharged into the environment (Lin et al., 2020). Heavy metals considered as the most important groups of water pollutants, are toxic (Benhima et al., 2008). The main sources of metal pollution, are natural (marine phosphates, weathering including erosion, non-volcanic soil, volcanic soil and volcanic activity) (Kumar et al., 2021) and anthropogenic sources (mining and smelting of metalliferous ores, burning of fossil fuels, wastes and sewage, pesticides and fertilizers (OECD, 2003) (Torbati & Keshipour, 2020; Hemalatha et al., 2021). Every year, the aqueous environment receives an average of millions tons of heavy metals as a consequence of human activities (Hu et al., 2016; Zhang et al., 2012).

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Cadmium and lead, among the most dangerous heavy metals, are frequently used in industrial processes (Volesky, 1991; Low & Lee, 1991; Chiban et al., 2011). They have no biological function (Chojnacka, 2010) and are harmful to aquatic ecological life, living organisms and human beings, even in low dosages (Fry et al., 1992; Zhang & Shao, 2013; Sheehan et al., 2014; Khan et al., 2015; Ayaz et al., 2020; Benhima et al., 2008). These heavy metals enters the food chain through potable water and sea foods, which endangers human life (Hemalatha et al., 2021).

Cadmium is a dangerous pollutant released from metal plating, ceramics, mining, electroplating, and the waste of used nickel-cadmium batteries (Chen et al., 2015; Martins et al., 2004; Ayaz et al., 2020), sewage sludge, cement industry, fuel combustion, power stations, protective plating on steel, Polyvinyl chloride (PVC) stabilizers, phosphate fertilizers, plastics, glass as a pigment, electrode material in nickel-cadmium batteries, mining activities and zinc smelting in various alloys (Huang et al., 2017; Malyan et al., 2019; Rehman et al., 2015; Sharaff et al., 2020; Singh et al., 2018; Kumar et al., 2021).

Cadmium is carcinogen (Kim et al., 2015; Kumar et al., 2021) and mutagenic (Beyersmann & Hartwig, 2008; Kumar et al., 2021). It can cause bone damage, hypercalciuria, hypertension, lung inefficiency, liver damage, renal dysfunction and neurological disorders in humans (Bernard, 2008; Cabral-Pinto et al., 2020; Kumar et al., 2021). Cd related health risk depends on its oxidation form and entry route (inhalation, ingestion) (Genchi et al., 2020). Acute exposure to Cd inhalation causes respiratory tract injury, interstitial pneumonia, pulmonary oedema and impairment of lung function. Chronic exposure of Cd will be seen in the bone, kidneys (proteinuria, renal stones, etc.) and causes Itai-Itai disease (Rahimzadeh et al., 2017; Kumar et al., 2021). Cd ingestion also affects the cardiovascular system, gastrointestinal tract, nervous system, kidneys and liver (Kumar et al., 2021).

Lead (Pb) is the second highest priority toxic heavy metal (Mal et al., 2021). Lead is of particular interest not only for its toxicity but also, by its widespread presence in the environment (Abdel-Halim et al., 2003).

Water resources are polluted with lead through various industries including electronics industry, metal-metallurgical industry, paint industry, oil refinery and mining industry (Mackay et al., 2013; Povedano-Priego et al., 2017; Mal et al., 2021), ceramics, paint, plastics, pesticide, automobiles, cement, and steel (Awual, 2017, 2019; Giri et al., 2022). The battery industry is considered as the major cause of water pollution (Roy et al., 2021; Badawy & Naguib, 2021).

Lead poisoning can cause various diseases which threaten human organs like brain and central nervous system, bony tissues development, gastrointestinal tract, kidney and liver (Zhang et al., 2019; Zhao et al., 2020; Hou et al., 2019; Badawy & Naguib, 2021). When discharged in the environment, the concentration of lead ion increases many folds and persist for a long time in soil, ground and surface water bodies. Thus, it enters in to the biological systems and affects living organisms. Its toxicity has severe effects on photosynthesis, nitrogen metabolism and cell division in plants (Wani et al., 2015; Giri et al., 2022).

Unlike organic pollutants, metals are non-biodegradable. The hypertoxicity of these metals leads to severe ecological effects. Thus, efficient processes have to be developed to eliminate them before their release into the environment (Benhima et al., 2008).

According to the World Health Organization (WHO), the maximal admitted concentration levels for cadmium and lead are 0.005 and 0.05 mg/L, respectively (Van der Leeden et al., 1990; Benhima et al., 2008; Dongre, 2020; Mal et al., 2021). Many techniques have been developed to treat heavy metal-polluted aqueous medium (Munter, 2013). For the elimination of cadmium and lead, a variety of physico-chemical methods exist. These methods include coagulation/flocculation, chemical precipitation, ion-exchange and adsorption (Chen et al., 2020; Wang et al., 2020; Torbati & Keshipour, 2020).

Usually, these strategies are too costly (High costs of equipment and high-operational costs) (Chen et al, 2015), and inefficient to reduce heavy metals concentration to the level required by water quality standards (Abdel-Halim et al., 2003). Moreover, they might produce a toxic waste that needs additional treatment (Saleh et al., 2020; Hu et al., 2016). New innovative technologies for water treatment are required (Chojnacka, 2010). Therefore, the research is oriented towards low cost and eco-friendly technology. Green technology as phytoremediation, has received a considerable attention (Sabreena et al., 2022) and is widely used. This environmentally friendly method has been successfully able to treat heavy metal polluted sites. Phytoremediation experiments using duckweed (*Lemna gibba*) have achieved high efficiency in assimilating large quantities of heavy metals (cadmium and lead) and nutrients (nitrate and phosphate) (Aggoun et al.,2018; Aggoun & Benmaamar, 2019). Many other experiences confirm the efficiency of *Lemna* sp. in the phytoremediation of heavy metals and different organic pollutants (Ali et al., 2016; Ekperusi et al., 2019).This phyto-process is successful but it has its limitation in heavy metal removal. The toxicity of these contaminants, can reduce duckweed biomass production or leads it to death (Satyakala & Jamil, 1992; Delgado et al., 1993; Miretzky et al., 2006).

In recent years, dried plants have been used in treatment of arsenate, nitrate, phosphate, cadmium and lead ions contaminated wastewaters (Chiban et al., 2011; Moussa et al., 2015).

The use of dead, dried aquatic plants, for metal removal as a biosorbent material has advantages. They are naturally renewable, and they process more quickly (Ighalo & Adeniyi, 2020).

In our previous works, experiments were conducted to explore the efficiency of the duckweed *Lemna gibba* in phytoremediation, for the removal of cadmium and lead in single and binary systems (Aggoun et al., 2018; Aggoun & Benmaamar 2019). The results showed an excellent uptake capacity of these toxic metals. In order to avoid the disadvantages of the phytoremediation by using live plant, in the present investigation, the adsorptive potential of the dead plant *Lemna gibba* as a low-cost natural material, was evaluated for Cd and Pb biosorption, individually and their mixtures. The effect of some factors such as contact time and adsorbent dose are also evaluated.

MATERIALS AND METHODS

Adsorbent plant material

At the beginning of spring, young fresh plants of the duckweed *Lemna gibba* were collected from a pond of north Algeria. The sampling site is located on Blida (36°36’50.7”N 2°49’48.0”E). These plants showed a greenish coloration indicating a good physiological state. Their selection was made due to their abundance.

The biomass was first rinsed with tap water, and then with distilled water, to obtain a clean biomass. This was then dried in the oven at 60°C to constant weight and then grounded by an electric mixer to produce a fine powder. Before starting the experiments, the ground dry powder of *L.gibba* is mechanically sieved to a suitable grain size (0.5 mm). The biomass powder was then prepared as described by Gardea-Torresdey et al. (1998). Briefly, 500 mg biomass sample was washed twice with 0.01 M HCl to remove any soluble biomolecules that might cause interference, and then cleaned with sterile distilled water. The sample was filtered and then dried at 65 °C for 48 h.

Chemicals

In this study, all chemical reagents used were analytical reagent grade (purity ≥99 %). Stock solutions of cadmium (Cd) and lead (Pb) were prepared by dissolving $CdCl_2$, H_2O and Pb (NO_3), in distilled water. The required concentrations of Cd and Pb solutions, were obtained by dilution with distilled water.

The initial pH value was adjusted by using 0.1 N of hydrochloric acid (HCl) and sodium hydroxide (NaOH). pH value was set at 4.0 ±0.5.

Before use, all the laboratory glassware used for experiments was cleaned with detergent, rinsed with tap water, soaked in 10% (v/v) nitric acid (HNO_3) and rinsed with distilled water.

Sorption experiments

Biosorption experiments were carried out in a thermostatic shaker at a temperature of (22±2) °C, and the agitation speed was kept constant (250 rmp). In glass flasks, a known mass (0.5 g) of dried and powdered *Lemna gibba*, was introduced in 100 mL of solutions contaminated with 0.1 or 1 mg/L of cadmium (or lead). The same procedure is followed in the binary mixture (Cd+Pb) where the combined concentrations of 0.1 mg/L or 1 mg/L of each metal are used.

Several authors have shown that acidic pH values are suitable for metal sorption (Halaimi et al., 2014; Chen et al., 2015). Thus, all sorption experiments were carried out at pH of 4.0 ±0.5

Effect of contact time

The effect of contact time on biosorption, was performed for 0.1 and 1 mg/L of Cd (Or Pb). The combined concentrations of 0.1 mg/L or 1 mg/L of each metal were used to evaluate the simultaneous contamination sorption tests. Thus, a sample of dead biomass (0.5g) was added to 100 mL of Cd or/and Pb solutions at room temperature and pH-value 4.

The flasks were shaken at 250 rpm for various periods (30, 60, 90,120, 180 and 240 minutes). At the end of each adsorption period, the biomass and the solution, contained in each flask, were separated from the solution by filtration using 0.45 µm acetate cellulose membranes. The filtrates were analyzed to determine the final Cd and Pb concentration in the samples. The removal efficiency and the biosorption capacity of Pb and/or Cd by the dead plants were reported by using Eq. (1) and Eq. (2) respectively:

$$\text{Removal efficiency; R \%} = \frac{(C_o - C_t)}{C_o} \times 100 \quad (1)$$

$$\text{Biosorption capacity; } q_t \text{(mg/g)} = \frac{(C_o - C_t)}{m} \times \text{V} \quad (2)$$

Where R% is the removal efficiency at each testing time, C_0 is the initial concentration of heavy metal (mg/L), and C_t is the concentration remaining in solution after each tested time of treatment (mg/L).

Effect of contaminant concentration

To evaluate the effect of Cd or Pb concentration on biosorption, two concentrations were tested (0.1 and 1mg/L). Thus, flasks containing 100 mL of medium and 0.5 g dead plant were contaminated with Cd and/or Pb at room temperature, shaking at 250 rpm, pH-value 4, and contact time corresponding to the determined equilibrium time.

Effect of biosorbent dose

Various weights of ground plants (0.10, 0.25, and 0.50 g) were added to flasks containing 100.0 mL of Cd and/or Pb solution (0.1, 1 mg/L or their mixtures) at room temperature, shaking at 250 rpm, pH-value 4, and contact time corresponding to the determined equilibrium time.

Heavy metals analysis

The final Cd and Pb concentrations were measured using inductively coupled plasma atomic emission spectroscopy (ICP-OES) (PerkinElmer, Optima 7300 V).

Analysis of Lemna gibba powder by Fourier transform infrared spectroscopy (FTIR)

The characteristics of the dead plants surface is probed by FTIR spectroscopy using a FTIR – 8201 PC, Shimadzu.
The ground dry powder of the duckweed, were pressed into slices with Bromide potassium (KBr). Slices were observed by FTIR before and after adsorption.

Kinetic adsorption models

Kinetic analysis was performed to give important information on the reaction's mechanism and pathway. It also provides data on the relationship between adsorption rate and the amount of pollutant adsorbed.
Adsorption kinetics provides a time-based measurement of adsorption uptake. The kinetic parameters give important information for designing and modelling adsorption processes (Pirzadeh & Ghoreyshi, 2014). Thus, biosorption data were analyzed with two kinetic models: pseudo-first order and pseudo-second order, according to Eq. (3) and Eq. (4) respectively (Elwakeel, 2010):

$$\mathrm{Log}(q_e - q_t) = \log q_e - (k_1/2.303)\ t \qquad (3)$$

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

Where k_1 is the pseudo first order rate constant (L/ min), q_e and q_t (mg/g) refer to the amount of metal ions adsorbed at equilibrium and at time t, while k_2 (g/(mg. min)) is the pseudo second order rate constant of adsorption.

RESULTS

Adsorbent characteristics

The process of adsorption is controlled by the molecular structure and the functional groups of the dried biomaterial (Saleh et al., 2020). As shown in table 1, the main functional groups of *Lemna gibba* prior to adsorption were the – OH and –NH stretching vibrations of amine and carboxylic groups, responsible for the broad peak at 3425.64. The peak observed at 2926.67 cm^{-1} is corresponding to the asymmetric stretching vibration of C–H bond (Sinharoy & Pakshirajan, 2019; Jain et al., 2015a; Li et al., 2017). The peak at 1653.83 cm^{-1} denotes amide stretching vibration of C=O group of carboxylic acid and the peak at 1420 cm^{-1} is due to the stretching vibration of C–H, whereas the peak at 1157.21 cm^{-1}can be assigned to the C–O stretching.
After Pb, Cd and Pb+Cd adsorption on the dead biomaterial most of the main peaks were shifted. The FTIR spectra of the powder dried plant loaded with Pb (0.1 mg/L), revealed peaks at 3445.44, 2920.21, 2369.76, 1647.08 and 1047.25 cm^{-1} and were 3425.64, 2922.47-2364.80, 1653.83 and 1047.27cm^{-1} at 1mg Pb/L.
The FTIR spectrum related to Cd adsorption on dried *Lemna gibba*, the peaks of the main functional groups are observed at the following wave numbers: 3396.29, 2930.86, 1649.64 and 1035.70 cm^{-1} at 0.1 mg/L and 3425.64, 2926.67, 1653.83, 1456.62 and 1054.99 at 1mg/L.
When Pb and Cd were fixed simultaneously on the biomass the principle peaks are located at 3551.43, 2926.67, 2369.99, 1544.74 and 1049.20 cm^{-1} in the mixture containing 0.1 mg/L of each metal. The peaks at 3287.25, 2933.47, 2373.18, 1651.73 and 1049.00 cm^{-1} are observed when dried *Lemna gibba* was loaded with 1mg/L each metal.

Effect of contact time

At constant pH-value (4.0±0.5) and ambient temperature (22°C), the effect of contact time on the retention of lead, cadmium and their mixtures, on dried *Lemna gibba* powder are depicted in figures 1 and 2.
The removal of Pb (Figure1a) increases significantly between 0 and 120 minutes, more slower between 120 and 180

Table 1. FTIR spectroscopy bands.

Cd or Pb concentration (mg/L)	Cd concentration (mg/L)		Pb concentration (mg/L)		Cd+Pb concentration (mg/L)	
0	0.1	1	0.1	1	0.1+0.1	1+1
3425.64 2926.67 1653.83 1420.0 1157.21	3396.29 2930.86 1649.64 - 1035.70	3425.64 2926.67 1653.83 1456.42 1054.99	3445.44 2920.21 1647.08 - 1047.21	3425.64 2922.47 1653.83 - 1047.27	3551.43 2926.67 1544.74 - 1049.20	3287.25 2933.47 1651.73 - 1049.0

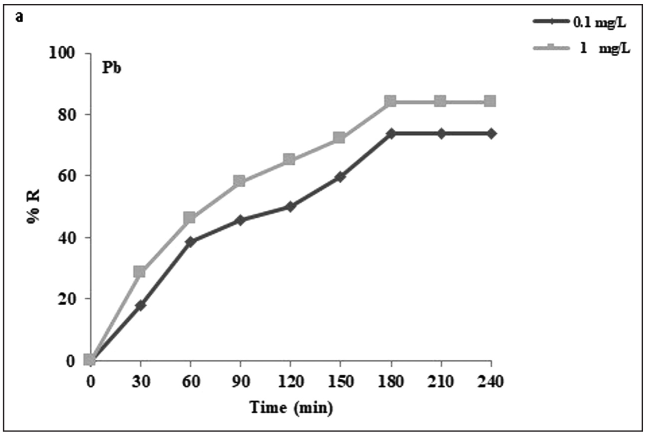

Figure 1a. Effect of time on Pb removal (%R) by dead *Lemna gibba*.

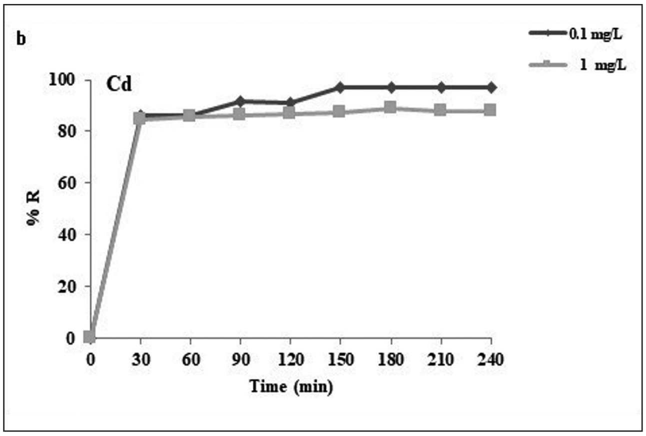

Figure 1b. Effect of time on Cd removal (%R) by dead *Lemna gibba*.

minutes and the percentage reduction remains unchanged from 180 to 240 minutes. The maximum values of 84.01% and 73.82% are obtained at 0.1 and 1 mg/L respectively. This suggests that during the second period, equilibrium is reached. Thus the equilibrium time is set at 120 minutes.

Regarding Cd (Figure 1b), the equilibrium is reached more rapidly (30 minutes) leading to a maximum removal of 93% and 88 % at 0.1 and 1 mg/L at the end of the experience.

On the other hand, the retention of Cd is reported to be larger than that of lead. Indeed, at 30 minutes, 28.66% of Pb is removed from the solution contaminated with 0.1mg/L (Figure 1a), while the removal rate of Cd reached 86.45% (Figure 1b). Similarly, in the presence of 1mg/L, Pb is removed from the solution at only 17.77%, while the percentage retention of Cd is 84.83%.

The amounts adsorbed of Pb and Cd by *Lemna gibba* powder increase with time to reach maximum values of 0.025 mg/g and 0.014 mg/g, respectively (Table 2), at 0.1 mg/L concentration. At 1 mg/L, amounts of 0.076 mg/g of Pb and 0.154 mg/g of Cd, are attached to the biosorbent.

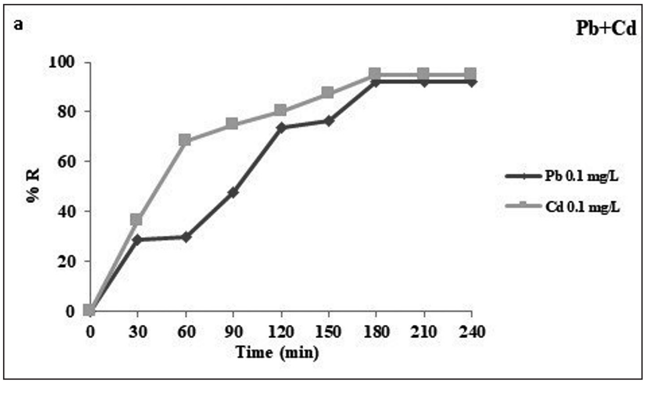

Figure 2a. Effect of time on Pb +Cd removal (%R) at 0.1 mg/L each metal by dead *Lemna gibba*.

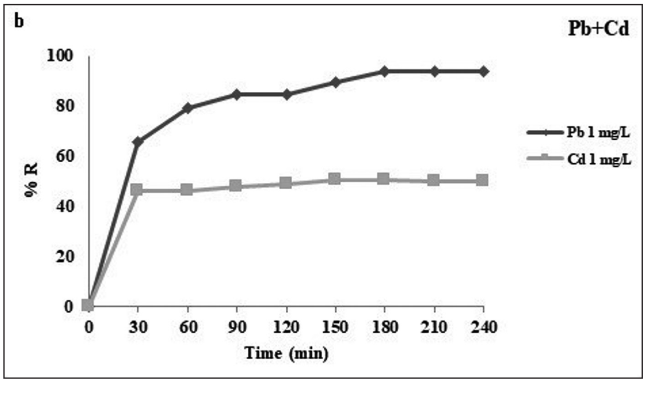

Figure 2b. Effect of time on Pb +Cd removal (%R) at 1 mg/L each metal by dead *Lemna gibba*.

Table 2. Fitting parameters of biosorption kinetic using Pseudo-first order and Pseudo-second order models**.**

		Pseudo-first order			**Pseudo-second order**		
Metal	**Concentration (mg/L)**	**R²**	**q_e (mg/g)**	**K_1(min^{-1})**	**R²**	**q_e (mg/g)**	**K_2(g.mg^{-1}. min^{-1})**
Pb $_{ind}$	0.1 1	0.920 0.974	0.0269 0.0772	0.015 0.011	0.982 0.982	0.035 0.095	0.357 0.132
Cd $_{ind}$	0.1 1	0.696 0.754	0.0085 0.041	0.020 0.026	0.999 0.999	0.019 0.154	6.333 3.926
Pb $_{mix}$	0.1 1	0.917 0.933	0.028 0.044	0.012 0.022	0.670 0.999	0.054 0.067	0.076 0.998
Cd $_{mix}$	0.1 1	0.979 0.822	0.017 0.046	0.016 0.027	0.989 0.999	0.023 0.096	0.774 1.888

In the Pb+Cd mixture, equilibrium is reached at 180 min in the presence of 0.1mg/L of each contaminant (Figure 2a). The removal of Pb and Cd is around 90%.

From 90min, the concentration of Pb and Cd in the solutions treated with 1mg Pb/L+ 1mg Cd/L changes slightly (Figure 2b). The maximum percentage of Pb removal is 86%, and that of Cd is close to 54%.

The amounts of Pb and Cd adsorbed by dried *Lemna gibba* powder from the solutions contaminated simultaneously by the two heavy metals are reported in table 2. The maximum amounts of Pb and Cd retained, are respectively 0.021mg/g and 0.017 mg/g in the mixture containing 0.1mg/L of each metal.

Lemna gibba powder retains a maximum of 0.063mg/g of Pb and 0.094 mg/g of Cd from the mixture contaminated with 1mg/L of each metal. From the results obtained, it appears that the quantities of Pb and Cd fixed on our biosorbent, from the solutions treated by the two metals individually, are very close to those retained from the mixtures.

It clearly appears that the retention of Cd and/ or Pb increases with the adsorbate-biosorbent contact time, to reach maximum values.

Table 3 – Biosorption capacities of Pb and Cd by dead *Lemna gibba*.

System	**C_0 (mg/L)**	**q_e (mg/g)**
Pb alone	0.1 1.0	0.025 0.076
Pb (Pb+Cd)	0.1 1.0	0.021 0.063
Cd alone	0.1 1.0	0.014 0.154
Cd (Pb+Cd)	0.1 1.0	0.017 0.094

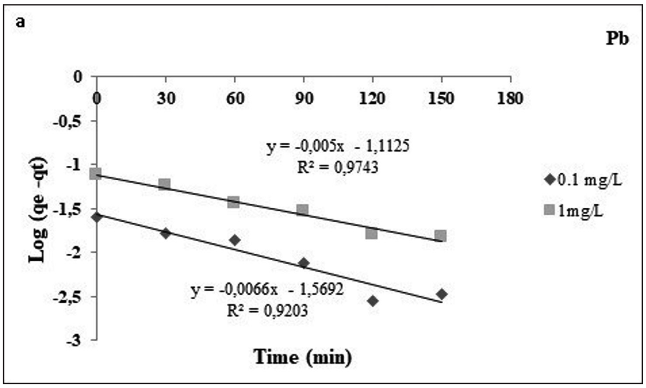

Figure 3a. Pseudo-first-order biosorption kinetics of Pbon dead *Lemna gibba*.

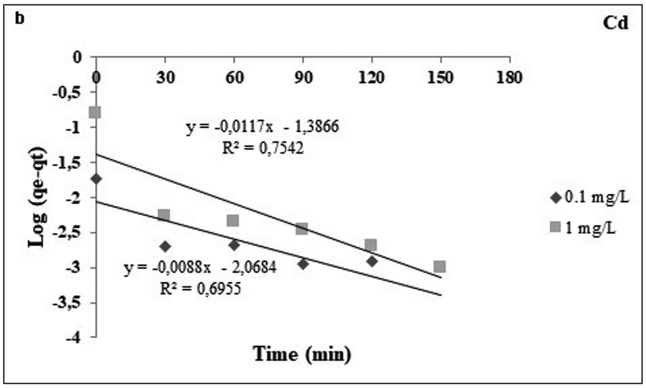

Figure 3b. Pseudo-first-order biosorption kinetics of Cd on dead *Lemna gibba*.

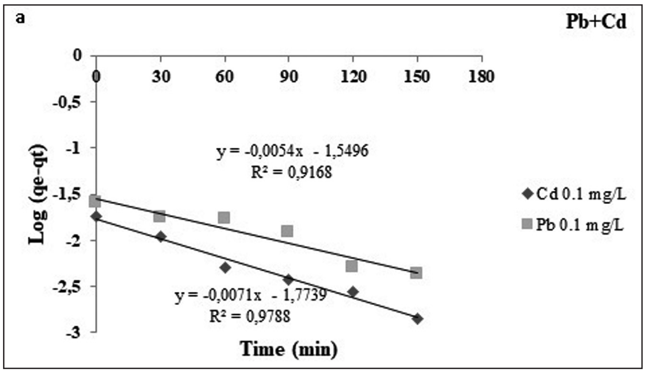

Figure 4a. Pseudo-first-order biosorption kinetics of Pb and Cd from the mixture Pb+Cd at -0.1 mg/L each metal on dead *Lemna gibba*.

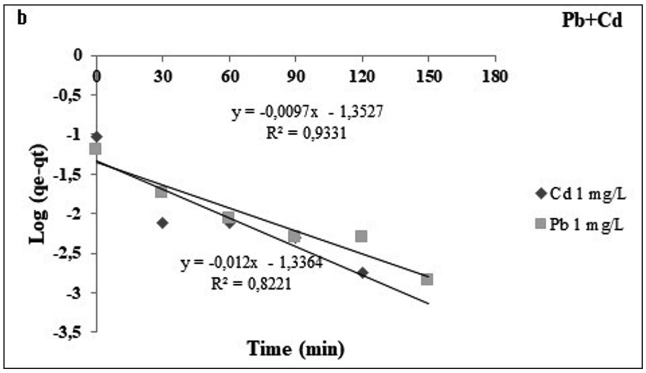

Figure 4b. Pseudo-first-order biosorption kinetics of Pb and Cd from the mixture Pb+Cd at 1 mg/L each metal, on dead *Lemna gibba*.

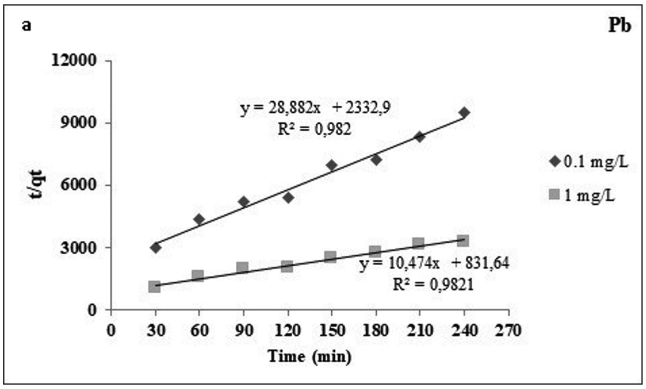

Figure 5a. Pseudo-second-order biosorption kinetics of Pb on dead *Lemna gibba*.

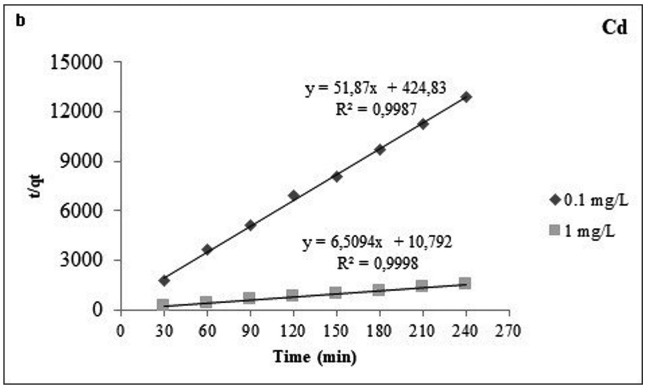

Figure 5b. Pseudo-second-order biosorption kinetics of Cd on dead *Lemna gibba*.

Modelling of biosorption kinetics

The linearized pseudo- first-order and pseudo-second order models of the sorption of cadmium and lead individually or combined onto dried *Lemna* powder at various initial concentrations are depicted in figures 3,4,5 and 6. The values obtained from the plots of the kinetic models, are shown in Table 3.

The pseudo second order model agreed better with the kinetics data of Pb sorption (Figure 5a) by dried *Lemna gibba* than the pseudo first order model (Figure 3a), with a high regression coefficient (0.982 at 0.1mg/L and at 1mg/L). Table 3 shows that the experimental q_e values (0.035 and 0.095) were quite near to the q_e values (0.027 and 0.077, respectively). In addition, the value coefficient R^2 of 0.999 suggested that Pb adsorption process follows second-order kinetics in the mixture comprising 1 mg/L of each metal (Figure 6.a). However, a straight line generated by plotting log (q_e-q_t) vs. t (Figure 4a) revealed that the pseudo-first-order equation suited the experimental findings well, yielding $R^2 = 0.917$ in the binary mixture with 0.1 mg/L each metal. The theoretical q_e values of 0.028 mg/g and the experimental data (0.021 mg/g) were almost identical (Table 3).

The pseudo-second order model for cadmium, either separately (Figure 5b) or in binary mixes (Figure 6b) showed that the correlation coefficients R^2, were found to be high (0.999). Furthermore, the experimental q_e measured are remarkably similar to those predicted by the plots (Table 3).

Effect of dry plant mass or dose

The results of the removal (R%) of lead, cadmium and their mixtures at the different amounts of the biosorbent, are shown in Figures 7a, b and Figures 8a,b.

For all the results, a considerable rise in the capacity of the biomaterial biosorption towards the contaminants is noticed with the increase of the biosorbent mass. Therefore, at the greatest mass value of the aquatic plant powder (0.5g), the retention percentages are ranging from 71% to 89%. Consequently, the optimal amount of biosorbent is 0.5 g/100mL. However, for the test performed with the mixture of 1mgCd/L+1mgPb/L, the maximum retention of 81.39% (Figure 8b) is reached for a mass of 0.25 g /100 mL.

It is well established that metal removal efficiency not only depends on the type of biosorbent but also on its quantity.

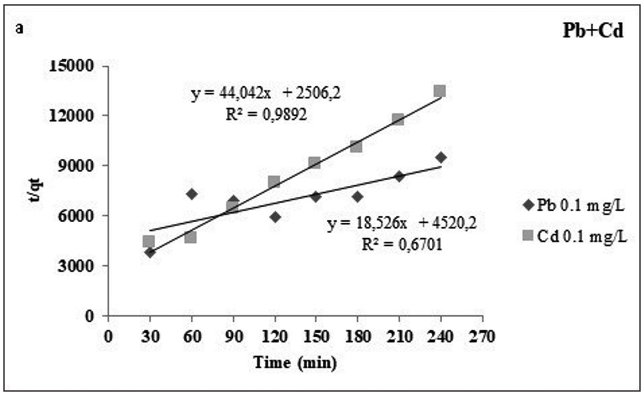

Figure 6a. Pseudo-second-order biosorption kinetics of Pb and Cd from the mixture Pb+Cd, on dead *Lemna gibba*.

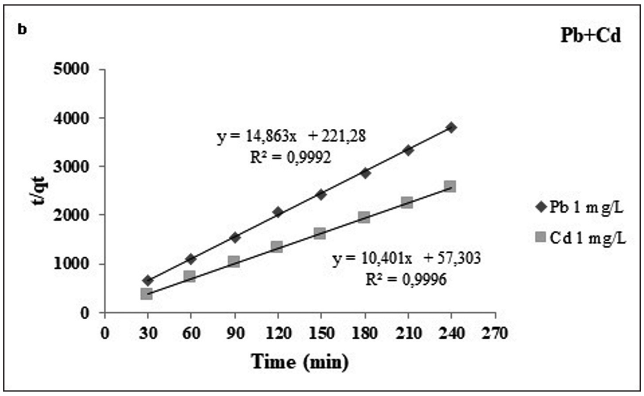

Figure 6b. Pseudo-second-order biosorption kinetics of Pb and Cd from the mixture Pb+Cd on dead *Lemna gibba*.

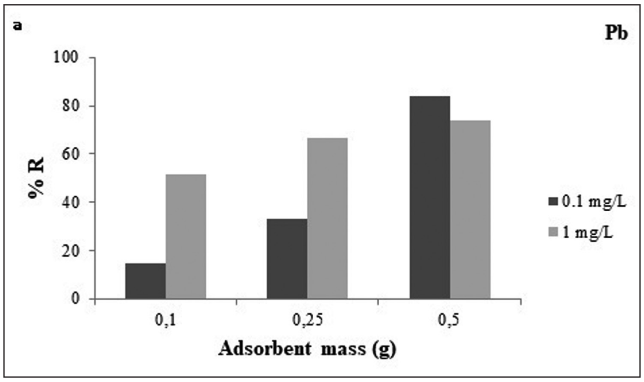

Figure 7a. Effect of the adsorbent mass on Pb removal.

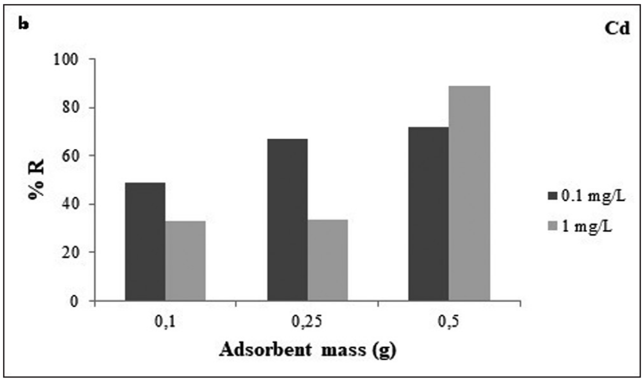

Figure 7b. Effect of the the adsorbent mass Cd removal.

Comparable findings have been recorded in the literature (Chen et al.; 2015).

Effect of contaminant concentration

Increasing the concentration of contaminants from 0.1mg/L to 1mg/L resulted in an increase in the amount of each metal adsorbed by dead *Lemna gibba* powder, either when contaminated individually or simultaneously with Pb and Cd. At equilibrium, Pb content increased from 0.025 mg/g to 0.075 mg/g and Cd content from 0.014 mg/g to 0.152 mg/g. Similarly, when the dead *Lemna gibba* is co-contaminated by Pb and Cd, the content of each metal also climbed when the concentration of each metal is raised from 0.1 to 1 mg/L.

DISCUSSION

Biosorption process depends on the molecular structure and the functional groups of the sorbent (Gusain & Suthar, 2017). FTIR analysis of dead *Lemna gibba* powder before and after adsorption of Pb, Cd and Pb+ Cd, demonstrated the presence of numerous functional groups involved in the adsorption of the metals either individually or in binary mixtures onto *Lemna gibba*.

Various significant peaks of the dried *Lemna gibba* in the spectrum are consistent with the previous work. O-H is the peak of 3700–3200 cm^{-1} which indicates polymeric compounds. The band around 2900 cm^{-1} was usually related to the C–H stretching vibration of CH_2 (Ghasemi et al., 2014). The peak of 1600–1300 cm^{-1} described the bonding of C-H is alkyl carbonate (C-OH) (Aichour & Zaghouane-Boudiaf, 2019; Singh et al., 2018; Ibrahim & Hamed, 2018; Saleh et al., 2020).

The contact time is crucial in adsorption for the removal of metals individually or in a combination (Chen et al., 2015). At equilibrium, the curves are in the form of a plateau showing that the biosorption of the solute is maximal.

The two stages of biosorption may be explained by considering that there are a set number of active sites in a system and that each active site can adsorb a single ion. Initial metal biosorption onto the biosorbent surface will be rapid, slowing down competition to reduce the availability of active sites (Li et al., 2008).

On the dried powder of duckweed *Lemna aequinoctialis*, the equilibrium between Cd ions and the adsorbent was reached within 180 minutes (Chen et al., 2015). In a similar study, Halaimi et al. (2014) found that Cd removal efficiencies on *Lemna gibba* powder were 50% and 60% at 0.1 et 1.0 mg/L respectively and equilibrium was achieved at 240 and 120 min.

In another study (Benhima et al., 2008), the initial stage of cadmium and lead adsorption onto dry plant microparticles is completed in no more than 30 minutes, with an uptake of around 81–87% for Cd and with up to 97% Pb ion removal. When Cd (II) and Pb (II) are adsorbed onto microparticles of dried Withania frutescens plant, The equilibrium is established in 60 minutes (Chiban et al., 2012).

Cd retention is larger than lead retention, probably due to the difference in the ionic radius of the two metals. Cd radius (0.95 Å) is smaller than Pb radius (1.19 Å), thus the motion of lead by diffusion, in the liquid is slower. Therefore, the transfer of Pb ions from most of the solution to the surface of the adsorbent is less than the transfer of Cd ions in aqueous solution. (Saleh et al., 2020).

The % adsorption of metal ions from Anza wastewater followed the order of Pb (II) > Cd (II). A similar trend has been noticed in the removal of divalent metal ions (Cu (II), Cd (II), Zn (II) and Pb (II)) by other plants (Benhima et al.,2008).

The use of *Lemna gibba* in the fresh state (phytoremediation) by Aggoun et al. (2018) resulted in Pb reduction of 57% at 1mg/L. In the binary mixture Cd+Pb (Aggoun & Benmaamar, 2019), the maximum reductions are 100 % of Pb and 41% of Cd at 0.1 mg/L each metal. The removal percentages are73 % of Pb and 27% of Cd in the mixture with 1 mg/L each metal.

In several studies examining the kinetics of metal adsorption onto various adsorbents, high correlations for the pseudo-second order model have also been discovered (Karthikeyan et al., 2005; Aydin & Askoy, 2009; Hu et al., 2011; Chen et al., 2015; Halaimi et al., 2014). This revealed that cadmium adsorption was the result of a chemical interaction. It also suggested that the rate of adsorption was related to the number of vacant sites.

The quantity of each metal absorbed by dead *Lemna gibba* powder increased when the pollutants' concentration was raised from 0.1 mg/L to 1 mg/L. Thus, the increased concentration of the two ions in the aqueous medium at the beginning of the biosorption process stimulates the diffusion of the ions from the liquid to the functional group of the biosorbent (Chen et al., 2015; Deng et al., 2016).

The electrostatic attraction type interactions between the positive charges of Pb and Cd and the negative charges of the biosorption sites situated on the surface of the dead *Lemna gibba* powder might possibly explain how lead or cadmium molecules attach to one another (Halaimi et al., 2014)

The amount of Pb and Cd that dried *Lemna gibba* powder absorbed increased when pollutants were added in concentrations ranging from 0.1 mg/L to 1 mg/L, whether Pb and Cd were added separately or concurrently. Cd content, increased from 0.014 mg/g to 0.152 mg/g and Pb concentration increased from 0.025 mg/g to 0.075 mg/g at equilibrium.

It is well established that metal removal efficiency not only depends on the type of biosorbent but also on its quantity. Comparable results are reported in the literature (Chen et al., 2015)

The observed improved lead and cadmium removal efficacy could be due to the vacant sites available for uptake of Pb and Cd species upon rise in biosorbent dose.

CONCLUSION

A common method for eliminating metal pollution and other hazardous elements from water is biosorption by dead dried plants. The natural material employed in this

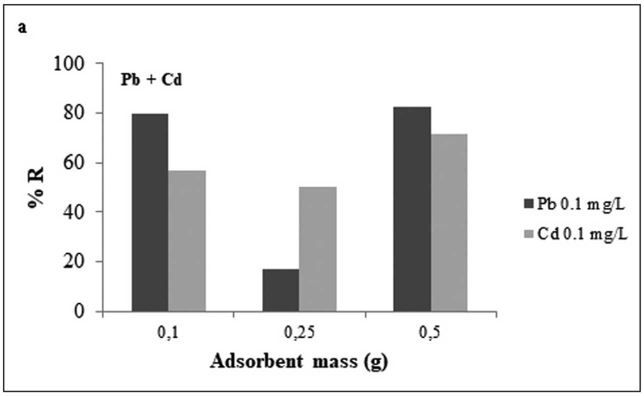

Figure 8a. Effect of the adsorbent mass on Pb +Cd removal at 0.1 mg/L each metal.

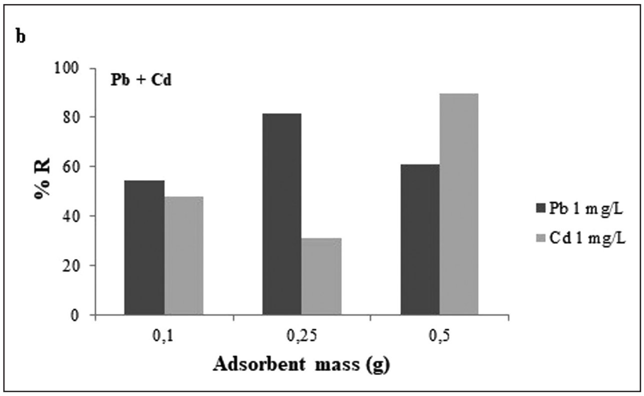

Figure 8b. Effect of the adsorbent mass on Pb +Cd removal at 1 mg/L each by dead *Lemna gibba*.

study is a good candidate as adsorbents in heavy metals removal approaches, considering the fact that this adsorbent is naturally ubiquitous and quite affordable. An effective and cheap adsorbent prepared from the dry biomass of *Lemna gibba* plant was successfully applied as biosorbent to remove highly toxic metals such as lead and cadmium either individually or in combination, from aqueous medium.

On the surface of the dead plant, FTIR analysis identified a number of relevant functional groups.

Biosorption of Pb and Cd on dead *Lemna gibba* surface increased with time and maximum adsorption achieved varied fom 73.82- 90% and 54- 93% respectively, either individually or in mixtures. The adsorption capacity of this material for cadmium and lead is of the same order of magnitude that has been found using other biosorbents or even higher than that when *Lemna gibba* was used in phytoremediation.

The pseudo second order model was found to suit Pb and Cd adsorption processes more closely than the pseudo first order model.

The use of this technology is expected to result in the efficient removal of hazardous metals, thus lowering the price of water purification with an ecological focus.

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