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Biosorption Of Pb(II) From Aqueous Solution Using Green Alga (*Ulvalactuca*) Biomass

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***Abstract*—The biosorption characteristics of Pb(II) ions from aqueous solution using the green alga (Ulva lactuca) biomass were investigated as a function of pH, equilibrium time, temperature, biomass dosage, and initial metal ion concentration. Langmuir and Freundlich models were applied to describe the biosorption isotherm of the metal ions by U. lactuca biomass. Maximum sorption was observed at a pH value of 5. Langmuir model fitted the equilibrium data better than the Freundlich isotherm. The monolayer biosorption capacity of U. lactuca biomass for Pb(II) ions was found to be 96.2 mg/g. The calculated thermodynamic parameters (ΔG°, ΔH° and ΔS°) showed that the biosorption of Pb(II) ions biomass was feasible, spontaneous and exothermic under examined conditions. The sorption kinetics was compatible with the pseudo-second order model for the systems investigated. SEM/EDX micrographs were determined before and after sorption.**

Keywords: Ulva lactuca; lead(II) ; biosorption; isotherm; thermodynamics; kinetics

I. INTRODUCTION

Manufacturing (plating, metallurgy, etc.) and mining industries are producing waste water streams contaminated with hazardous metal ions. International regulations are becoming more and more drastic concerning the level of metal discharge in the environment. Lead is a metal that was widely used for many basic applications in industry for the manufacturing of batteries [1], paints and so on. Lead may have a cumulative effect in the organism accumulating throughout the food chain.

For these reasons a great attention has been attached for the removal of heavy metal ions from industrial wastewater in order to reduce the global impact of these metals on the environment. Different processes can be used for the removal of lead from aqueous streams, including precipitation [2], solvent extraction [3, 4], solvent impregnated resins [5, 6], ion exchange resins [7], or membrane processes. However, these processes generally face economical (expensive treatments for low-concentration effluents), technical (difficulty to raise the regulation levels) or environmental (sludge production, loss of solvent) constraints that limit their application. Biosorption that consists in using materials of biological origin for the sorption of target solutes has been proposed as an alternative to convention ion exchange and chelating resins or activated carbon.

Among the biological materials marine algae otherwise known as seaweeds have been reported to have high metal binding capacities due to the presence of polysaccharides, proteins or lipid on the cell wall surface containing functional groups such as amino, hydroxyl, carboxyl and sulphate, which can act as binding sites for metals [8]. The green alga, *Ulva lactuca*, is particularly useful in these respects because of its wide distribution along the shores of Algeria [9].There is no extensive study on the biosorption of Pb(II) using this algal biomass in literature. In addition, this new material was chosen as biosorbent in this study due to being of its natural, renewable and thus low-cost biomass.

The present study investigates the potential use of *U. lactuca* biomass *for* sorption of Pb(II) ions from aqueous solution. The influence of pH, sorbent dose and initial concentration of metals on biosorption were investigated. Experimental sorption isotherms were fitted to the Langmuir and Freundlich models. The biosorption mechanisms were also investigated in terms of thermodynamics and kinetics.

I. MATERIALS AND METHODS

A. Materials

Samples of the biomass were collected from the West Mediterranean Sea coast of Algeria and washed several times using deionized water to remove extraneous and salts. They were then dried in an oven at 60 °C for 48 h. The dried algae biomass was chopped, sieved and the particles with an average of 0.5mm were used for biosorption experiments. Metal ions stock solutions were prepared from analytical grade $PbCl_2$ (Fluka AG, Switzerland) and were further diluted as required with deionized water produced in the laboratory, then acidified at pH 5.

B. Characterization of materials

With a Scanning Electron Microscope (SEM), the topography of the samples was observed using secondary electron flux, while the backscattered electrons were used for the identification and localization of heavy metals at the surface of the materials (by phase contrast). SEM-EDAX facilities were used for the analysis of specific zones at the surface of biosorbent. The dry sorbent was analyzed using an Environmental Scanning Electron Microscopy (ESEM) Quanta FEG 200, equipped with an OXFORD Inca 350 Energy Dispersive X-ray microanalysis (EDX) system.

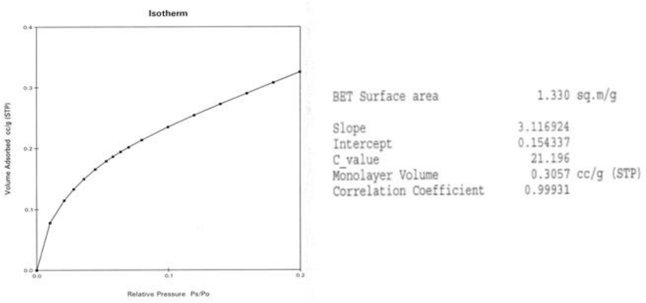
C. Sorption experiments

Initial pH was varied from a value of 2 to 6 by addition of concentrated hydrochloric acid or caustic soda as required to a suspension prepared by mixing 20 mg of biosorbent with 25 mL of solution of Pb(II) or Cd(II) (100 ppm). The resulting suspension was agitated magnetically for 48 h at 20 °C and the final pH value recorded. Sorption isotherms were tested at 20 °C by contact of 25 mL (volume, V) of metal solution (C_0, concentration ranging between 10 and 500 mg Pb L^{-1}) with 20 mg of biosorbent (amount m, g) for 48 h (under 200 rpm agitation). After filtration (though a 1.2 µm pore size filter membrane) residual metal concentration (C_{eq}, mg Pb L^{-1}) was determined by ICP-AES (Jobin-YvonActiva M, Jobin-Yvon, Longjumeau, France). The mass balance equation ($q= V(C_0-C_{eq})/m$) served to determine the sorption capacity (q, mg Pb g^{-1}). In order to examine the adsorption controlling mechanism, pseudo-second-order kinetic models [10, 11] were applied to experimental data of the studied metal ions. Kinetic experiments were conducted for different initial quantities (0.5 g, 1.0 g and 1.5 g) of biomass to which identical volumes of Pb(II) solutions (100 g/L) were added in stirred flasks for 72 h at a speed of 200 rpm, a pH of 5, and a temperature of 20 °C. Solution samples of 4 mL were withdrawn at regular intervals, filtered and the metal ions concentration measured. Similar tests were performed by varying the initial concentration of the metal ions (50, 100 and 200 mg/L).

II. RESULTS AND DISCUSSION

A. Characterizations of Biosorbent

The small surface area found (Fig. 1), around 1.33 m^2/g, is compensated by the numerous and complex functional groups present on the algal surface.



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BET Surface area                 1.330 sq.m/g

Slope                          3.116924
Intercept                      0.154337
C_value                          21.196
Monolayer Volume                 0.3057 cc/g (STP)
Correlation Coefficient        0.99931
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Fig. 1: Isotherme of BET Surface area

The algal biomass analysis by Scanning Electron Microscopybefore metals uptake is shown in Figure 2. The micrographs show the appearance of grains on biosorbent after biosorption whereas they were absent before the biosorption. The SEM observed grains were also characterized by energy dispersive X-rayspectrometry: EDX. Figure 3 shows the signals of Pb(II) on the surface of biosorbent.

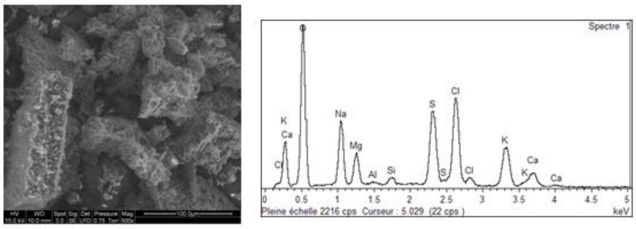

Fig. 2: SEM/DEX of algal biomass before lead uptake.

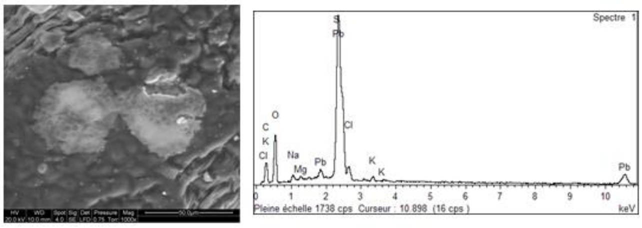

Fig. 3: SEM/DEX of algal biomass after lead uptake.

Table 1 and 2 summarize the chemical composition of biosorbent used before and after biosorption of lead ions. There is a marked decrease, especially Na and K ions whose values fall from 8 to less than 1 in percentage by mass, And also for the Mg which passes from 3.5 to 0.4 and the Ca of 2.8 to a total disappearance, which could be explained by an exchange phenomenon.

TABLE 1: Chemical composition of algal biomass (SEM/DEX) before lead uptake.

Element	O	**Na**	**Mg**	Al	Si	S	**Cl**	**K**	**Ca**	Total
% Mass	51.05	**8.70**	**3.59**	0.21	0.81	9.83	**14.78**	**8.22**	**2.81**	100.00
% Atomic	67.07	**7.96**	**3.11**	0.16	0.61	6.44	**8.77**	**4.42**	**1.47**	

TABLE II: Chemical composition of algal biomass after lead biosorption (SEM/DEX).

Element	C	O	**Na**	**Mg**	S	**Cl**	**K**	**Pb**	Total
% Mass	23.69	26.66	**0.96**	**0.40**	6.38	**1.54**	**0.76**	**39.62**	100.00
% Atomic	47.53	40.15	**1.00**	**0.39**	4.79	**1.05**	**0.47**	**4.61**	

B. pH effect

The effect of hydrogen ions concentration on biosorption of heavy metals has been the subject of many studies which showed the importance of this parameter on the solubility of the metal ions as well as on the ionization of the fixing sites [12].

Algal biomasses contain high content of carboxyl groups from mannuronic and guluronic acids on the cell wall polysaccharides, which suggests that the biosorption process could be affected by changes in the solution pH [13]. The effect of pH on the biosorption of Pb(II) ions onto *U. lactuca* biomass was studied by changing pH values in the range, 2–6 and the results were presented in Fig. 4. The metal ions uptake was affected by the pH of the medium as it increased with solution pH increase. Increased positive charge (protons) density on the sites of biomass surface at low pH values (pH 2–4) restricted the approach of metal cations as a result of repulsive force. In contrast, when the pH value increased, biomass surface was morenegatively charged and the biosorption of metal ions with positive charge (Pb^{2+}) was reached maximum around pH 5. The sharpest increase in maximum capacity (qe) was obtained between pH values of 3 and 5, corresponding for the latter pH value to capacities of 90 mg/g. Decrease in biosorption at higher pH (pH > 5) is due to the formation of hydroxilated complexes of the metal ions and their competition with the active sites, and as a consequence, the retention would decrease. To avoid metal precipitation biosorption tests were not conducted beyond this maximum value of pH [13].

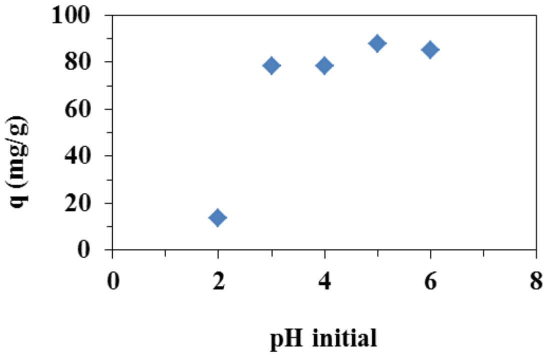

Fig. 4: Initial pH effect on Pb removal

(Ci=100 mg/l, 20 °C, 200 rpm, particle size: 250–500 µm).

C. Biosorption isotherms

The equilibrium biosorption isotherm which gives the capacity of the biosorbent at a fixed temperature is an important physicochemical quantity for evaluating a biosorption process [14]. The adsorption isotherms considered here are: Langmuir and Freundlich adsorption isotherms. The Langmuir model assumes that biosorption occurs at specific homogeneous sites on the biosorbent and is used successfully in many monolayer biosorption processes. The linear form of this model can be written as follows: $\frac{Ce}{q} = \frac{Ce}{qm} + \frac{1}{qm*b}$ [15], where *q* is the equilibrium metal ion concentration on the biosorbent (mg/g), *C*e is the equilibrium metal ion concentration in the solution (mg/L), *q*m is the monolayer biosorption capacity of the biosorbent (mg/g), and *b* is the Langmuir biosorption constant (L/mg) relating the free energy of biosorption. Fig. 5 indicates the linear relationship between the amount (mg) of Pb(II) ions sorbed per unit mass (g) of *U. lactuca* biomass against the concentration of Pb(II) ions remaining in solution (mg/L). The coefficients (R^2) were found to be 0.97 for Pb(II) biosorption, indicating that the biosorption of the metal ions onto *U. lactuca* biomass fitted well the Langmuir model. In other words, the sorption of metal ions onto *U.lactuca* was taken place at the functional groups/binding sites on the surface of the biomass which is regarded as monolayer biosorption. From equation of linear form of Langmuir model we obtain the Langmuir parameters: the *b* value was found as 0.12 L/mg for Pb(II) ion. The maximum biosorption capacity (*q*m) was found to be 96.2 mg/g. In addition, Table 3 presents the comparison of biosorption capacity of *U. lactuca* biomass for the Pb(II) with those of various biomasses in literature [16,17,18]. The biosorption capacity of *U. lactuca* biomass for this metal ion is higher than that of the majority of other biomasses given in Table 3. Therefore, it can be noteworthy that the *U. lactuca* biomass has important potential for removal of Pb(II) ions from aqueous solution.

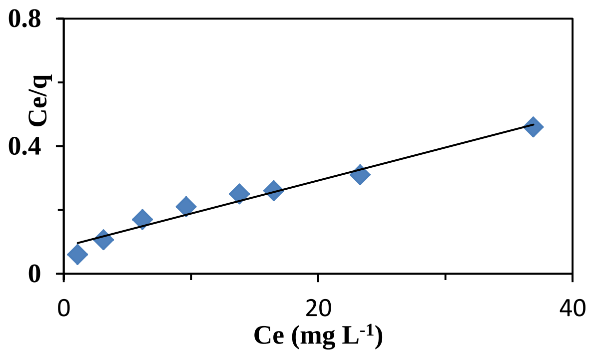

Fig. 5 Langmuir isotherms for Pb(II)biosorption at (pH=5 and 20 °C).

TABLE 3: Monolayer biosorption capacity(mg/g) of some algal biomasses taken from literature

Biomass	q_m(mg/g)	Ref
U. lactuca	96.2	Present study
Laminaria japonica	5.2	1
Ulva spp	54.3	2
Cladophoraspp *Spirogyra spp*	46.51 90.91	3
Dry algal mixture	281.8	4
Ulvaspp	34.7	5
H. fusiformis *L. japonica* *U. pinnatifida*	136.99 192.31 181.82	6
U. lactuca(activated carbon)	83.3	7

The Freundlich model has been widely used to describe adsorption experiments. In this model, the uptake of metal ions occurs on a heterogeneous adsorbent surface. It is assumed that the stronger binding sites are occupied first and that the binding strength decreases with the increasing degree of site occupation [19, 20]. The linearized form of Freundlich equation is given by:

$$logq = logk + \frac{1}{nlogq} \quad (3)$$

where the parameters k and n can be obtained from the intercept and slope of the plot of logq versus log Ceq. The Fig. 6 shows the Freundlich isotherms obtained for the biosorption of Pb(II) ions onto *U. lactuca* biomass using Eq. (3). The values of *k* and *n* were found to be 2.77 and 0.81. The 1/*n* values were between 0 and 1, indicating that the biosorption of Pb(II) onto *U. Lactuca* biomass was favorable at studied conditions. In addition, compared to the R^2 values, 0.970, with that obtained from the Langmuir model, it can be remarkably noted that the Freundlich isotherm model is better fitted the equilibrium data.

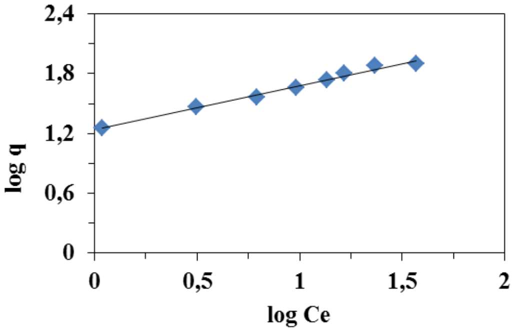

Fig. 6: Freundlich isotherm plots for biosorption of Pb(II) onto*U. lactuca*

biomass (biomass dosage: 20 g/L; contact time: 60 min; pH: 5; temperature: 20 ◦C).

D. Kinetic Studies

Kinetic studies are performed to explain the mechanisms involved in the biosorption of micropollutants. The process of biosorption of metals in the liquid phase by porous solids is controlled by external diffusion, the intraparticle diffusion and the step of adsorption [21].

- *Effect Of Initial Concentration*

FIG.7 shows a good fixation at low concentration (10 mg / L) with a Ce / C_O ratio of less than 0.2 and an equilibrium time of more than 50 minutes, which is explained by the availability of functional sites responsible for biosorption. With increasing initial concentration (from 50 to 200mg / L) the Ce/C_0 ratio increases (and thus the fixed amount decreases) and the equilibrium time decreases due to rapid saturation of the binding sites (less 25mn for the initial concentration of 200mg / L).

The initial metal concentration constitutes a significant drivingforce allowing the metallic mass transfer between the aqueous and the solid phases [22].

According to [23, 24], the biosorption was decreased with increasing the metal concentration. This behavior was attributed to the fact that, initially, all binding sites on the biomass surface were vacant resulting in high metal biosorption at the beginning. Afterthat, with increasing metal concentration, the biosorption of metal was decreased because of a few active sites were available on the surface of the algal biomass.

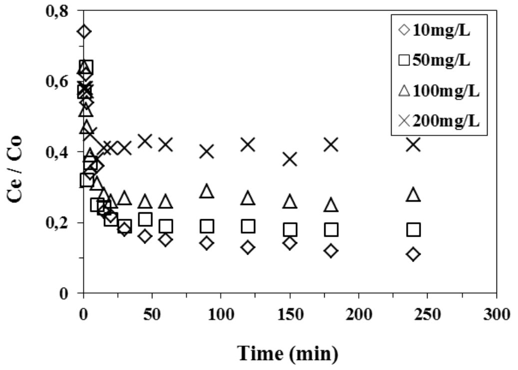

Fig.7: Biosorption kinetics of Pb removals as a function of initial concentration.

• *Effect Of Algal Biomass Dose*

The algal dose effect on the not fixed quantity of lead is shown in fig.8. This quantity relative to the initial amount (Ce/C_0) is about 0.2 for quantities of biomass of 1 and 1.5 g and 0.3 for 0.5 of biomass. For the three quantity of biomass the maximum of lead is biosorbed during the first 20 minutes.

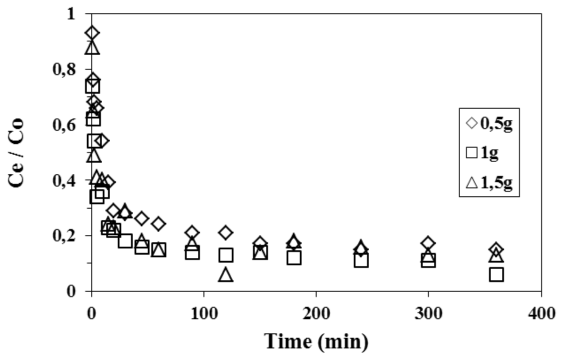

Fig. 8: Biosorption kinetics of Pb removals as a function of biosorbent dose

E. Temperature Effect On Sorption Isotherms And Thermodynamic

Study

The effect of temperature is shown in fig.9 and thermodynamic parameters obtained from the following equations are indicated in table IV.

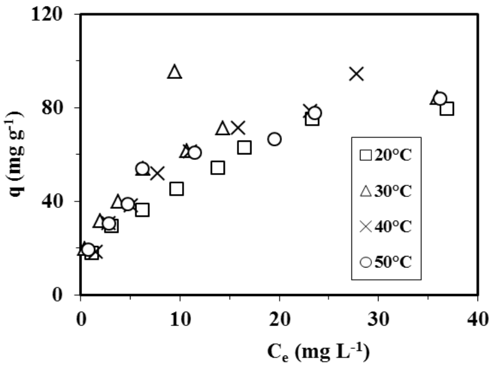

Fig.9: Effect of temperature on Pb(II) sorption isotherms at pH 5.

Increasing the temperature slightly improves the sorption capacity of lead ions at pH 5, as shown in fig 9 which confirms previous studies using the same biomass [25, 26, 27]. It is noteworthy that the affinity coefficient (which is proportional to the initial slope of the curve) decreased, in a general way (a dispersion was observed in the distribution of the values), with increasing temperature. The isotherms were modeled using the Langmuir equation (Table IV). To gain insight to the mechanism involved in the adsorption process, thermodynamic parameters for the present system were calculated. The relationship between equilibrium constant (Kc) and enthalpy (ΔH^0) is the thermodynamic basis for predicting the change in equilibrium constant with temperature. The value, ΔG^0, gives information about the spontaneity of the adsorption process [28, 29, 30].

$$\Delta G_0 = - RT\ln K_c \quad (1)$$

$$\Delta G_0 = \Delta H_0 - T\Delta S_0 \quad (2)$$

$$\ln Kc = \frac{\Delta S0}{R} - \frac{\Delta H0}{RT} \quad (3)$$

TABLE IV: Effect of temperature on sorption isotherms for Pb(II)

binding on algal biomass—Langmuir parameters.

T (°C)	q_m (mg Pb g^{-1})	b (L mg^{-1})	R^2 (%)
20	96.2	0.12	97
30	91.7	0.3	97
40	117.7	0.11	98
50	93.3	0.19	98

The negative values of ΔG^0 with temperature increases indicate that the reaction is spontaneous and that the biosorbent have a great affinity for Pb(II) uptake. On the other hand, the positive value of the entropy indicates an increase in the randomness at the solid and solute interface (Table V). A positive change in the entropy may be explained by some structural changes in the adsorbate and the adsorbent [31, 32, 33].

Table V—Thermodynamic data for the sorption of Pb(II) using Algal biomass (pH 5).

T° K	ΔG^0 (kJ/mol)	ΔH^0 (kJ/mol)	ΔS^0 (kJ/mol)
293	-30.15	+1.58	102.9
303	-31.18		
313	-32.21		
323	-33.24		

III. CONCLUSION

Previous work has shown that algal biomass can be used as a bosorbent for Pb(II). The present work investigated the potential use of *U. lactuca* biomass *for bio*sorption of Pb(II) ions from aqueous solution studying the effect of several effects on Pb(II) fixation like pH, sorbent dose and initial concentration The experimental biosorption data for metal ion fitted reasonably well the Langmuir model. The maximum uptakes obtained at a pH of 5 for Pb(II) were 96.2 mg.g^{-1}.

The biosorption kinetics for the metal ions studied at different initial solutions concentrations, suggesting a chemical process for the mechanism of biosorption between the metal ions and the sorbent. For the three quantity of biomass the maximum of lead is biosorbed during the first 20 minutes. The effect of temperature on biosorption does not show a real change in lead uptake. The study of biosorption isotherms at different temperatures allowed evaluation of this process thermodynamically, the positive value of entropy indicates that the biosorption of lead increases the randomness (disorder) at the interface between the solid and solution.

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