Journal of Applied Sciences

ISSN 1812-5654

Journal of Applied Sciences 7 (14): 1864-1872, 2007
ISSN 1812-5654

Removal of Heavy Metal Ions (Pb^{2+}, Cu^{2+}) in Aqueous Solutions by *Pterygota macrocarpa* Sawdust

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Abstract: The purpose of this study is the use of *Pterygota macrocarpa* sawdust as adsorbent for lead and copper removal into aqueous acid solutions. The results showed that the rate of removal is better for particle sizes lower than 0.5 mm, in the metal solutions at pH 3. The Langmuir, Freundlich and Temkin isotherms studies were allowed to determine the maximum capacity of adsorption of the sawdust; it is 115.61 and 24.02 mg g^{-1} for the lead and cooper removal, respectively. This study also showed that the metal ions removal is accompanied by a releasing of K^+, Ca^{2+} and Mg^{2+} in the metal solutions. This use could constitute a way of valorisation of the sawdust, a main waste of the wood industry.

Key words: Heavy metals, removal, sawdust, lead, copper, Langmuir, Freundlich and Temkin isotherms

INTRODUCTION

Environmental pollution has become a reality impossible to circumvent in developing countries when taking into account its fast growth noted during these last years. This growth is due to the industrialization policy and the space extension very little controlled consequence of a very strong demographic growth. This situation contributes in a dominating way to a permanent environmental pollution. To face this situation is thus a question of valorisation of waste or treatment of the ecosystems by effective and low-cost techniques. Unfortunately the majority of the technical and technological solutions tested elsewhere are still not transposable. Thus, it is necessary for those which are transposable to be adapted to the local conditions.

The research tasks concerning the use of sawdust or by-products of the food industry for the purification of wastewater are numerous (Fiset *et al*., 2000; Karthikeyan *et al*., 2002; Ho *et al*., 2002; Meunier *et al*., 2003a, b; Sèbe *et al*., 2004; Larous *et al*., 2005; Taty-Costodes *et al*., 2005; Safarik *et al*., 2005; Montes *et al*., 2006; Abdel-Ghan *et al*., 2007). Some of them showed the performance of these adsorbents for the recovery of metals ions in aqueous solutions. Recently, Meunier *et al*. (2003a, b) showed that the cocoa shells present interesting properties of metals removal in leachates of soil decontamination with a strong capacity for lead. The removal of the metal ions by the sawdust of *Epiciea* was developed by Marin (2001). During this study, the author optimized the conditions of elimination of toxic metal ions (cadmium, copper, nickel, lead and zinc) contained in worn water. The use of clays in the field of the liquid waste processing was also the several research task objects (Ferrandon *et al*., 1998; Kugler *et al*., 2002). Ferrandon *et al*. (1998) presented a study with a well provided references and showed the role of some clays in the wastewater treatment for the elimination of viruses, coliforms, humic substances, greases and oils. Microporous materials of vegetable origin such as the activated carbon, because of their adsorbent capacity, are largely used for the extraction of chemical substances in aqueous and gas phases (Desjardins, 1990; Avom *et al*., 2002; Singh *et al*., 2006; Gueu *et al*., 2006, 2007; Britton Bi *et al*., 2006).

In this study, we present the results of metal ions removal in acid aqueous solutions by *Pterygota macrocarpa* sawdust. Indeed, the sawdust is the main waste of wood industry. Taken as such, the sawdust constitutes an important problem for the wood industry and can present significant environmental and human health risks. Large sawdust layers exist but only very little are used (only in breeding). The mode of elimination of these by-products usually used is the incineration. This practice, although sometimes necessary, only makes it possible to get rid of some, without drawing any advantage from their physical properties. Moreover, the incineration does nothing but amplify the phenomenon of the effect of greenhouse.

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This study aims at contributing to the industrial processing wastewater containing of metal ions before their natural discharge and especially to propose a method of valorisation of the principal waste of the wood transformation.

MATERIALS AND METHODS

The basic aim of the experiment is to evaluate the removal of lead and copper in aqueous solutions by using the sawdust of *Pterygota macrocarpa* as an adsorbent.

Adsorbent: Sawdust used in this study comes from the sawmill *Tropical Bois* of Adzopé (in South-Eastern Côte d'Ivoire). It acts of the sawdust of Koto wood, 30 m height tree, one still finds in the forest zone of Côte d'Ivoire. Its scientific name is *Pterygota macrocarpa* and forms part of the great family of *Sterculiacees* (Aubréville, 1959).

The sawdust was dried during two days at 60°C and then sieved. Two particle sizes, less than 0.5 mm (sawdust A) and ranging between 0.5 and 1 mm (sawdust B) have been directly used without any pre-treatment, as an adsorbent for the removal of lead (II) and copper (II) ions from aqueous solutions.

Adsorbate solutions: The tests of removal of Cu^{2+} and Pb^{2+} ions were carried out using synthetic solutions obtained starting from nitrate salts of copper ($Cu(NO_3)_2.3H_2O$) and anhydrous lead ($Pb(NO_3)_2$). Nitrate salts were selected because of the low chelating capacity of NO_3^- with respect to the metal ions (Marain, 2001). The solutions of ions at pH 2 and 3 were prepared using double distilled water, acidified with concentrated nitric acid. The solutions are prepared at the experiment day.

Continuous experiment procedures: The experiment was carried out in a column packed with sawdust. A glass column of 25 cm length and 5 cm internal diameter has used. The column was packed with a known dose (10, 15 and 20 g) of sawdust. A percolation of 100 mL of an aqueous solution of metal ions with a certain concentration (100 and 300 mg L^{-1}) allows to obtain the samples. After 10 min of contact time, the supernatant is filtered through a 0.45 μm membrane filter to remove particulates and is used as sample. After each test, the column content was evacuated and rinsed with double distilled water, then with a nitric acid solution of pH 3 then again, with double distilled water. The column was packed with a new sawdust dose for other experiment. The metal content of the samples was determined by an atomic absorption (Varian AA 20 model) in an air-acetylene flame. The value of the pH was measured by a pH-meter Hannan HI-98150 with the supernatant. The results are expressed in terms of percent removal of metal ion and capacity of the sawdust for metal ions, according the following relationships:

$$\%\,\text{removal} = \frac{C_0 - C_1}{C_0} \times 100 \tag{1}$$

$$\text{Capacity}\,(\text{mg g}^{-1}) = \frac{C_0 - C_1}{m} \times V \tag{2}$$

Where:

C_0 = The initial concentration of metal ions solutions (mg L^{-1}).
C_1 = The concentration in metal ions of the filtrate.
V = The volume of metal solution (L)
m = The weight of adsorbent (g).

Batch experiment procedures

Adsorption kinetics: The kinetics of metal ions removal is capital. Indeed, it makes it possible to determine the time necessary to obtain the maximum capacity of ions removal. Batch experiments have been carried out by shaking 3 g of the sawdust with 200 mL of lead (II) and copper (II) aqueous solutions (pH 3) of 300 mg L^{-1} concentration at a 30±0.2°C. The suspension is agitated at 300 rpm during variable time ranging 5 and 90 min. At the end of each time, the mixture is filtered and a sample of filtrate is analyzed by means of the atomic absorption. The measurement of the pH is carried out with the remaining filtrate.

Sorption equilibrium studies: Batch sorption tests were done out at 30±0.2°C at particle size less than 0.5 mm. At each test, 3 g of sawdust was mixed into 200 mL solution of different initial metal ion concentrations ranging from 30 to 300 mg L^{-1}. The contact time is determined by the kinetics study, it was 45 min. But during the tests we took 60 min in order to have a steady balance. After 60 min of agitation, each mixture is filtered and the metal content of the filtrate determined. The results are expressed in terms of the Langmuir, Freundlich and Temkin isotherms.

RESULTS AND DISCUSSION

Continuous experiments: The metal ions in aqueous solutions are fixed at the adsorbing support by varying the operating conditions. These variations concerned the particle size of the sawdust and the pH of the solution for the experiments carried out with lead. While copper, we varied the dose of the sawdust B at pH fixes then carried out cycles with solutions of concentration 100 and 300 mg L^{-1}. Thereafter, we tried to understand the mechanism of the removal of the metal ions by the sawdust by a kinetic study and three adsorption isotherms.

Effect of particle sizes: The effectiveness of the sawdust of *Pterygota macrocarpa* with respect to the Pb^{2+} ions was evaluated with lead ion solution (100 mg L^{-1}) at pH 2 and 3. It can be observed that the removal of lead ion by sawdust increases while the particle size of adsorbent decreases (Fig. 1). Thus with this size (less than 0.5 mm) of the sawdust, the maximum rates (62.67%) at pH 2 and 63.99% (pH 3) were obtained. As for the sawdust B, this rate varies from 40.21% (pH 2) to 44.31% for the solution with pH 3.

Thus, adsorption seems to be the dominating phenomenon in the retention of the Pb^{2+} ions. In more adsorption, it exist an ionic exchange. Indeed, the solution with pH 2 being richer in protons (0.01 mol L^{-1} of H^{+}), those occupy a considerable sites on the sawdust, thus limiting the retention of the Pb^{2+} ions. As for the solution to pH 3 (0.001 mol L^{-1} of H^{+}), the competition is reduced, which would facilitate the access of the sites to the Pb^{2+} ions, thus leading to a rate of retention higher than in the case of the solution with pH 2. However, for higher values of the pH, the formation of metal hydroxides by precipitation of the ions, their quantity in the solution will be limited.

Figure 2 shows copper removal by the sawdust B at different pulp density values at pH 2 from aqueous solutions containing 100 mg L^{-1} (S_1) and 300 mg L^{-1} (S_2). It can be observed that the retention of the copper by sawdust increases while the pulp density value increases. It is clear that adsorption increased from 14.16 to 18.34% for the solution of 100 mg L^{-1} and 14.9 to 31.18% for the solution of 300 mg L^{-1} with the amount of sawdust from 10 to 30 g L^{-1}. This may be attributed to the increase of the sites of adsorption. Indeed, the more solution is concentrated; the more the ions approach the available sites. Also, the possibility of a competition with the protons in solution is slowed down contrary to what would occur in the case of the lower concentrations. The solution of 300 mg L^{-1} being concentrated in Cu^{2+}, the competition would be favourable to the Cu^{2+} ions and thus would limit the fixing of the protons (Meunier *et al*., 2003b; Marin, 2001).

Effect of cycles: One hundred milliliter of solution is treated several times by a fixed amount of sawdust. Several cycles are carried out with each dose (10, 15 and 20 g) of the sawdust B and a volume of 1 mL is taken after

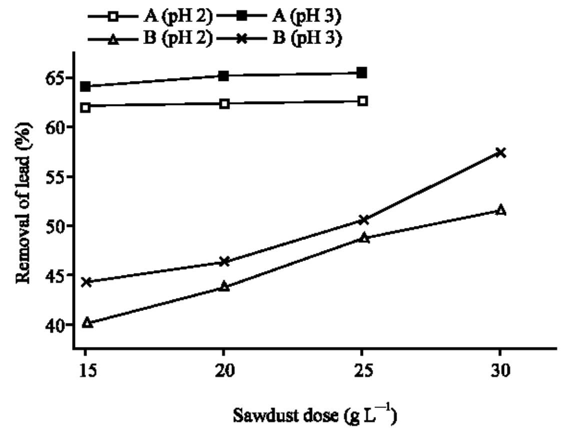

Fig. 1: Rate of Pb^{2+} ions retention according to the sawdust dose. A represents the sawdust A, B the sawdust B and between bracket the initial pH of the effluent

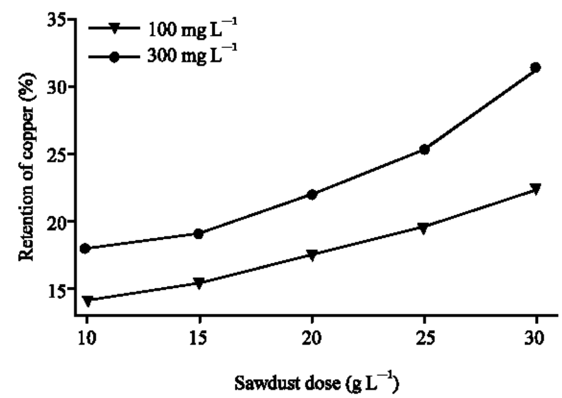

Fig. 2: Rate of Cu^{2+} ions removal according to the sawdust dose

each cycle. Figure 3 and 4 showed the effect of the cycles on the copper ions removal by the sawdust B. The repeated circulation of the same solution through the same layer of sawdust allows a better purification of the effluent. This elimination is more often accompanied by a phenomenon of adsorption-desorption which starts after the adsorbent saturation. The best rates of retention are obtained at 3rd cycle for the solution of 100 mg L^{-1} and between 2nd and 3rd cycles for the solution of 300 mg L^{-1}. With the effluent of 100 mg L^{-1}, the average of purification lies to 2.18 mg g^{-1} while in the solution of 300 mg L^{-1}, the rate of retention varies from 9.04 to 9.13 mg g^{-1}. Saturation is quickly reached for the solution of 300 mg L^{-1} because of its strong metal concentrations. The continuation of the operations beyond the cycle of saturation immediately involves a fall of the rate of retention of copper and could be explained by the copper desorption in the solution.

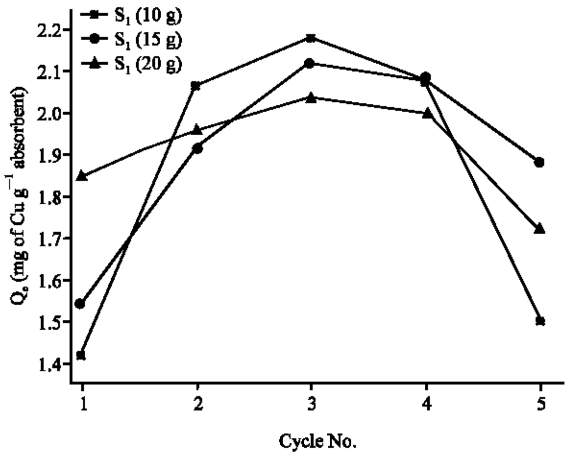

Fig. 3: Amount of Cu^{2+} removal according to the number of cycles (solution 100 mg L^{-1}). S_1 (X g): copper solution of 100 mg L^{-1} treated by X g of sawdust

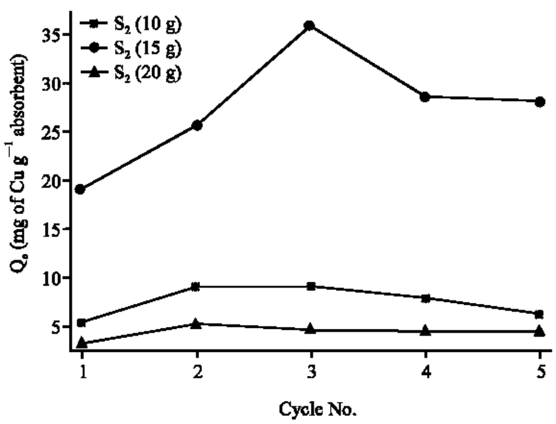

Fig. 4: Amount of Cu^{2+} removal according to the number of cycles (solution 300 mg L^{-1}). S_2 (X g): copper solution of 300 mg L^{-1} treated by X g of sawdust

Discontinuous experiments

Effect of pulp density: The adsorption of Pb^{2+} and Cu^{2+} at pH 3 and 30±0.2°C from three aqueous solutions containing 300 mg L^{-1} of metal ions (Fig. 5). Two solutions containing each metal ions and the mixture containing 300 mg L^{-1} of each ion. The solutions were treated by the sawdust with a mean particle size lower than 0.5 mm. The adsorption capacity values of metal ions indicate that the best value is obtained when the adsorbent dose is 15 g L^{-1}. This value is used for the other experiments.

Adsorption kinetics: The kinetic of the Pb^{2+} and Cu^{2+} removal was carried without solutions containing 300 mg L^{-1} of monometallic, then with a solution

Table 1: Amount of the elements (K^+, Ca^{2+}, Mg^{2+}) released in water at pH 3 as function as the contact time

Time (min)	K^+ (mg L^{-1})	Ca^{2+} (mg L^{-1})	Mg^{2+}(mg L^{-1})
5	52.23	17.59	40.20
10	53.05	17.82	42.03
25	53.30	16.89	43.38
20	52.83	18.09	45.58
30	53.15	19.27	49.90
45	53.10	22.54	51.63
60	54.15	23.19	51.93
90	54.60	23.74	52.08

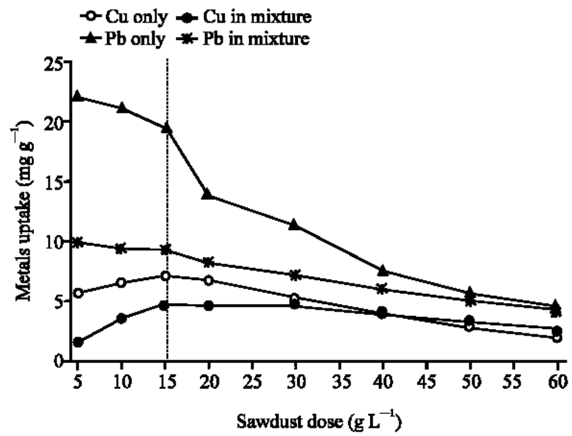

Fig. 5: Effect of pulp density of the adsorbent on metals ions removal

containing both ions at 300 mg L^{-1} of each ion (mixture). The solutions are treated by 15 g L^{-1} of the sawdust with a mean size of 0.5 mm. These results show a selectivity of the sawdust with respect to lead in both solutions with a strong rate of removal of 83% (16.6 mg g^{-1}) in lead solution and it clearly shows that equilibrium is attained only after 30 min. In the mixture, the maximum rate of lead removal (51% corresponding to 10.20 mg g^{-1}) is obtained after 45 min (Fig. 6). While the copper, the rate of removal is rather weak; 29% (5.8 mg g^{-1}) in the solution of copper and 23% (4.6 mg g^{-1}) in the mixture.

In these solutions, the pH varies very little; from 3 to 4.6 in lead solution, 3 to 3.4 with the solution of copper and 3 to 4.3 in the mixture. This variation of the pH is due to a reduction of the metal ions in solution but especially to a leaching of ions (K^+, Ca^{2+} and Mg^{2+} measured during this study) in the solution by the sawdust. The study of the removal of the cations on the sawdust by comparison with the behaviour of the sawdust in the metal ions solutions and of water at pH 3 is allowed us to observe ion-exchange phenomena. The values of the ions (K^+, Ca^{2+}, Mg^{2+}) leached in water and in metals solution are shown in Table 1 and 2, respectively. It shows that magnesium is the element which is abundantly leached. In the nitric acid solution of pH 3, the sawdust leaching

Table 2: Amount of elements (mg L^{-1}) released into the metal ions solutions

Time (min)	pH3 Lead ions solution K^+	Ca^{2+}	Mg^{2+}	Copper ions solution K^+	Ca^{2+}	Mg^{2+}	Mixture (Pb and Cu) K^+	Ca^{2+}	Mg^{2+}
5	2.41	2.49	15.20	0.84	22.01	18.49	4.75	10.50	19.05
10	4.14	4.23	14.57	0.20	25.73	23.11	4.20	17.49	24.45
15	4.10	6.60	14.17	0.20	30.16	23.21	3.73	20.70	25.80
20	3.48	5.51	13.37	1.22	30.71	21.21	5.25	21.37	25.30
30	4.04	4.88	09.33	0.92	30.45	17.64	5.21	26.07	22.20
45	2.90	3.64	08.20	1.15	30.43	16.61	5.40	24.45	21.02
60	3.31	1.46	08.47	0.50	30.96	16.49	4.55	24.35	21.10
90	3.81	2.36	09.97	0.47	31.86	16.88	4.05	24.55	21.22

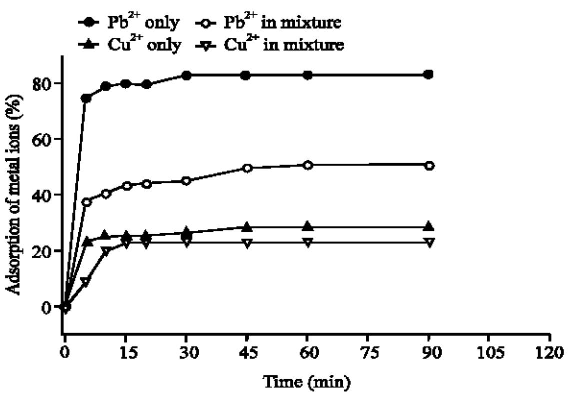

Fig. 6: Effect of contacting time of the metals removal by *P. macrocarpa* sawdust

(Table 1) is done according to the following order: $K^+ > Mg^{2+} > Ca^{2+}$. In the lead solution alone of pH 3, this order becomes:

$Mg^{2+} >> Ca^{2+} \approx K^+$, while the copper solution it is: $Ca^{2+} > Mg^{2+} >> K^+$ and in the mixture it becomes: $Mg^{2+} \approx Ca^{2+} >> K^+$.

Although the elimination of the Cu^{2+} and Pb^{2+} is accompanied by leaching of strong quantity of magnesium, there would existed other elements whose exchange supports the metal ions removal; it would be as potassium for lead and calcium for copper.

Adsorption isotherms: The study of sorption isotherm is fundamentally important in the determination of the maximum capacity of the adsorbent. In order to investigate the capacities of lead and copper sorption onto *P. macrocarpa* sawdust-the Langmuir, Freundlich and Temkin models were analyzed.

Langmuir isotherm: The Langmuir isotherm has been used by many researchers to study the sorption of many heavy metals. The model assumes uniform energies of adsorption onto the surface and no transmigration of adsorbent in the plane of the surface (Krishnan and Anirudhan, 2003; Rachakornkij *et al*., 2003; Nabizadeh, 2005). The Langmuir equation is given:

$$Q_e = \frac{K_L Q_o C_e}{1 + K_L C_e} \tag{3}$$

Where:

Q_e = The amount metal ions adsorbed per unit mass of adsorbent (mg g^{-1}).

Ce = The metal concentration in solution at equilibrium (mg L^{-1}).

K_L = Langmuir isotherm constant related intensity of adsorption.

Q_o = Langmuir monolayer sorption capacity.

The linear form of Langmuir isotherm is given by Eq. 4:

$$\frac{1}{Q_e} = \frac{1}{K_L Q_o Ce} + \frac{1}{Q_o} \tag{4}$$

The parameters were obtained by fitting the experimental data of the plot of $1/Q_e$ versus $1/C_e$ for lead and copper removal at different concentrations. The values of Q_o and K_L (Langmuir constants) were obtained from the intercept and slope of Eq. 4.

The plots of Eq. 4 and 3 are shown in Fig. 7 and 8, respectively. The values of Langmuir parameters and the coefficient of correlation (R^2) are shown in Table 3. The maximum capacities of metal ions removal are as lead concerning 115.61 and 24.02 mg g^{-1} for the copper. The apparent energy/intensity of sorption given by the value of K_L showed that the energy of adsorption is not very favourable to Cu^{2+}. The maximum adsorption capacities obtained by Langmuir isotherm model were used to estimate the specific surface area (S) of adsorbent using following equation (Ho *et al*., 2002):

Table 3: Isotherm constants for metal ions sorption onto *Pterygota macrocarpa* Sawdust

	Langmuir			Freundlich			Temkin		
Metal	K_L	Q_o (mg g^{-1})	R^2	K_F	1/n	R^2	K_T	$b_{T\,kJ/mol}$	R^2
Lead	0.0023	115.61	0.9989	0.3201	0.9242	0.9953	0.1080	0.3414	0.9645
Copper	0.0609	24.02	0.9736	0.0368	1.0365	0.9025	0.0465	0.8147	0.9771

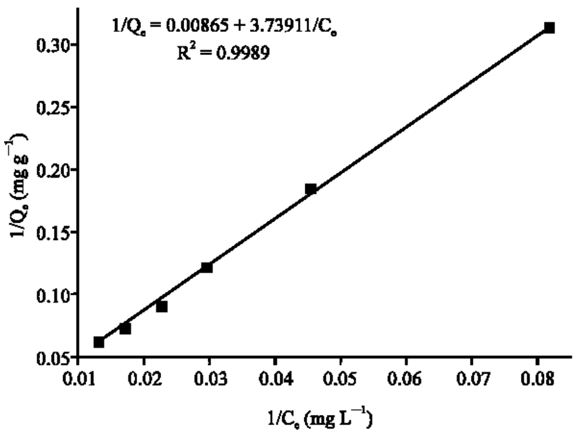

Fig. 7: Langmuir isotherm of Lead adsorption onto *P. macrocarpa* sawdust

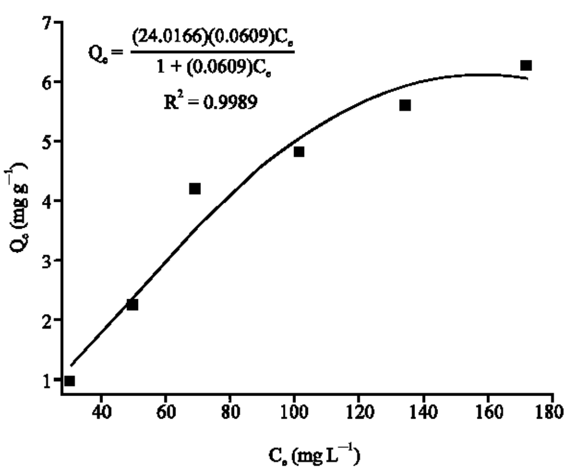

Fig. 8: Langmuir isotherm of copper adsorption onto *P. macrocarpa* sawdust

$$S = \frac{Q_o N A}{M} \quad (5)$$

Where:

- Q_o = The monolayer adsorption capacity (mg g^{-1}).
- N = Avogadro number (6.022 10^{23} mol^{-1}).
- A = The sectional area of ions, 5.56 $Å^2$ for Pb^{2+} and 1.58$Å^2$ (Cu^{2+}).
- M = Molecular weight of metal, 63.55 g (Cu) and 207.2 (Pb).

Table 4: Specific surface areas for Pb^{2+} and Cu^{2+}

Metal	Q_o (mg g^{-1})	S (m^2 g^{-1})
Lead	115.61	18.69
Copper	24.02	3.60

The values obtained have been shown in Table 4. The differential removal of the two ions may be described to the difference in their ionic radius. Indeed, the smaller the ionic radius or area, the greater is tendency to hydrolysed leading to reduced sorption (Horsfall and Spiff, 2005a).

Freundlich isotherm: The Freundlich model is an indicator of extent of heterogeneity of the adsorbent surface. For this model, the concentration of solute in solution at equilibrium (C_e) and the amount of solute adsorbed being Q_e are connected by the following equation:

$$Q_e = K_F C_e^{1/n} \quad (6)$$

where, K_F and n are the Freundlich constants and represent the adsorption capacity and intensity of adsorption, respectively. The linear form is given by the following equation:

$$LnQ_e = LnK_F + \frac{1}{n} LnC_e \quad (7)$$

The plots of LnQ_e versus LnC_e for lead and Q_e versus C_e for copper are shown in Fig. 9 and 10, respectively. Values of K_F and 1/n shown in Table 3 were determined from the intercept and slope of the plot LnQ_e versus LnC_e and from non-linear regression of Eq. 6. The Freundlich isotherm constants show that the affinity of the sawdust with respect to lead compared to copper. Indeed, according to Tahar *et al*. (2004), the adsorption is in conformity with the isotherm of Freundlich if $n > 1$.

Temkin isotherm: The linear form of the Temkin isotherm model as shown in Eq. 8 (Horsfall and Spiff, 2005b):

$$Q_e = \frac{RT}{b_T} LnK_T + \frac{RT}{b_T} LnC_e \quad (8)$$

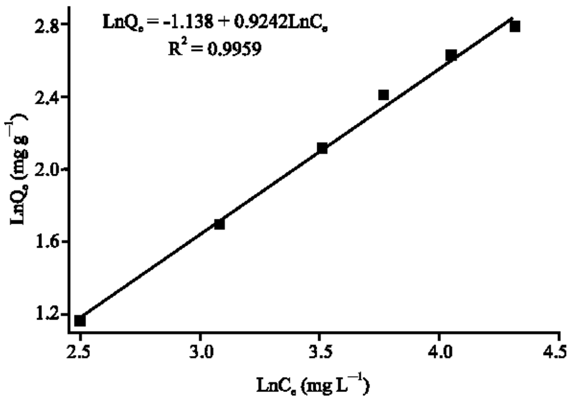

Fig. 9: Linear Freundlich isotherm of lead removal by *P. macrocarpa* sawdust

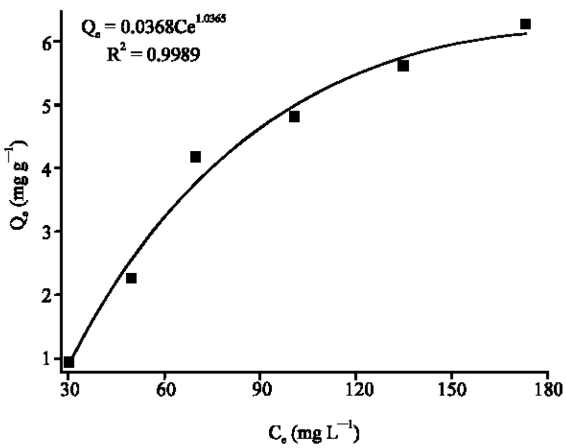

Fig. 10: Freundlich isotherm of copper removal by *P. macrocarpa* sawdust

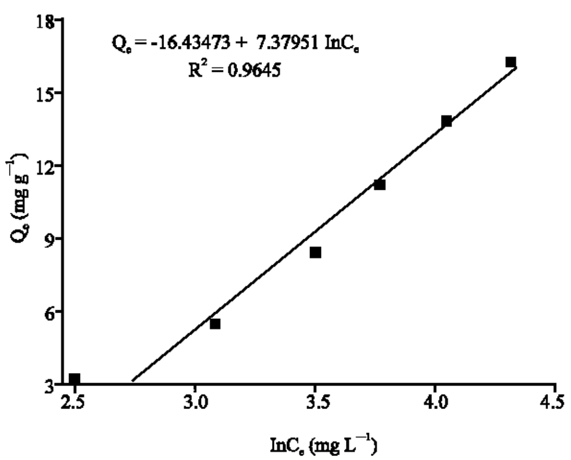

Fig. 11: Temkin equilibrium isotherm model for lead sorption onto *P. macrocarpa* sawdust

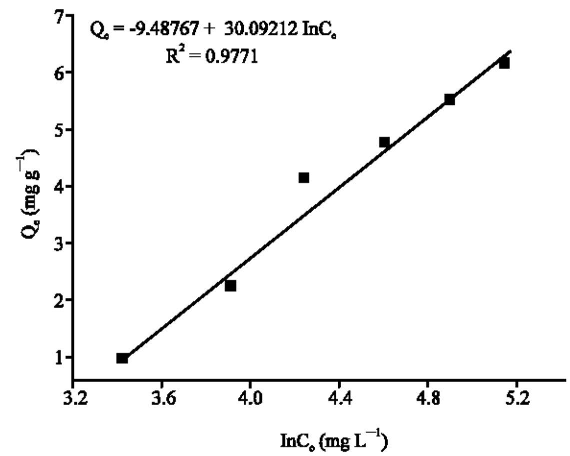

Fig. 12: Temkin equilibrium isotherm model for copper sorption onto *P. macrocarpa* sawdust

Where, b_T indicates the adsorption potential of the adsorbent and K_T is the Temkin constant. Values of K_T and b_T were calculated from the intercept and slope of the plot of Qe versus LnCe (Fig. 11 and 12) and the Temkin isotherm parameters is given in Table 3. The low values obtained, indicates weak interactions between metal ions and adsorbent, supporting an ion-exchange mechanism.

The coefficients of correlation (R^2) listed in Table 3 indicated that Langmuir and Temkin isotherms fitted the adsorption data for metal (lead and copper) better than Freundlich isotherm. As the lead concerning, Langmuir and Freundlich models give the best values, while the copper the Langmuir and Temkin isotherms exhibit the better models.

CONCLUSIONS

The principal aim of the present research was to evaluate the potential use of the sawdust of *Pterygota macrocarpa* as an adsorbent for of metal ions removal in aqueous solutions. The results showed that lead and copper can be removed at a short contact time, 30 min for lead and 45 min in the case of copper. The selectivity order for metal ion towards the studied sawdust is $Pb^{2+} > Cu^{2+}$. It was noted that ionic area has an influence in metal ions loading on the sawdust. The maximum adsorption capacity of this sawdust is 115.61 mg g^{-1}, for lead and only 24.02 mg g^{-1} as far as copper is concerned. The adsorption of the metal ions is carried out by a mechanism of ion-exchange, implying mainly magnesium but also potassium and calcium.

The results reveal that this sawdust is particularly efficient to remove lead and, to lesser degree, copper from acid solutions. Thus, the by-product of *Pterygota macrocarpa* transformation constitutes a promising adsorbent for environment protection.

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