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Applicability of *Moringa oleifera* Lam. pie as an adsorbent for removal of heavy metals from waters

Affonso C. Gonçalves Junior[1], Ana P. Meneghel[1], Fernanda Rubio[1],
Leonardo Strey[1], Douglas C. Dragunski[2] & Gustavo F. Coelho[1]

ABSTRACT

This study evaluated the efficacy of moringa seeds (*Moringa oleifera* Lam.) as an adsorbent material for removing toxic heavy metals such as cadmium, lead, and chromium from contaminated solutions. The effect of the adsorbent mass was investigated at two pH conditions (5.0 and 7.0). The optimized conditions were 0.300 g of adsorbent at pH 5.0, used for the isotherms construction, and linearized according to Langmuir and Freundlich models. Results showed that cadmium adsorption was similar in both the models used. For lead, the Freundlich model had the best adjustment and chromium was better adjusted by the Langmuir model. It was concluded that the adsorbent was effective in the remediation of solutions containing cadmium, lead and chromium, thus, its use as sustainable alternative material is feasible, since it has low cost, does not need a previous treatment and it is a byproduct.

Key words: adsorption, isotherms, remediation

Aplicabilidade da torta de *Moringa oleifera* Lam. como adsorvente para remoção de metais pesados de águas

RESUMO

Este estudo objetivou avaliar a eficácia do uso da torta de moringa (*Moringa oleifera* Lam.) como material adsorvente dos metais pesados tóxicos cádmio (Cd), chumbo (Pb) e cromo (Cr) de soluções contaminadas. Nos testes cinéticos foram variadas as massas do adsorvente em duas condições de pH (5,0 e 7,0). As condições otimizadas foram pH 5,0 e massa de 0.300 g de adsorvente, utilizados para a construção das isotermas e linearizadas conforme os modelos de Langmuir e Freundlich. Realizou-se a determinação dos metais por espectrometria de absorção atômica. Os resultados mostraram que houve semelhança em ambos os modelos utilizados para a adsorção do Cd. Para o Pb, o modelo de Freundlich apresentou o melhor ajuste e, para o Cr, houve melhor ajuste pelo modelo de Langmuir. Conclui-se, com base nos resultados obtidos, que o adsorvente foi eficaz na remediação de soluções contendo Cd, Pb e Cr e, assim, é viável a utilização desse adsorvente como material alternativo sustentável, pois apresenta baixo custo, não necessita de tratamento prévio e se trata de um coproduto.

Palavras-chave: adsorção, isotermas, remediação

[1] UNIOESTE, Rua Pernambuco 1777, CEP 85960-000, Marechal Cândido Rondon, PR. Fone: (45) 3284-7924. E-mail: affonso133@hotmail.com; mel_meneghel@hotmail.com; fernandarubiofoz@hotmail.com; leonardostrey@yahoo.com.br; gfc_faramir_@hotmail.com
[2] UNIPAR, Praça Mascarenhas de Moraes, s/n, CEP 87502-210, Umuarama, PR. Fone: (44) 3621-2828. E-mail: dcdragunski@unipar.br

INTRODUCTION

A major problem today is the environmental pollution coming largely from the improper management of pesticides, low-quality water used in the irrigation process and indiscriminate disposal of industrial or domestic waste, which may cause the accumulation of substances that can be toxic to plants and may become dangerous to animals and human beings if they come in the food chain (Camargo et al., 2006).

The water contamination by heavy metals has been a major concern for researchers and government agencies involved with the pollution control (Oliveira et al., 2001), mainly by toxic heavy metals, harmful contaminants due to their ability of retaining and accumulating in the human body (Reddy et al., 2010).

Some metals are used in the biological metabolism and thus may be considered essential, such as copper (Cu), zinc (Zn), nickel (Ni) and chromium (Cr), which at higher concentrations can become toxic. However, lead (Pb) and cadmium (Cd) are considered toxic even at trace levels (Gonçalves et al., 2009).

An efficient remediation alternative of natural resources contaminated with toxic heavy metals is the adsorption process, which can regulate the mobility and bioavailability of pollutants in solution (Araújo et al., 2002).

The removal of heavy metals from effluents involving adsorption on organic and inorganic materials may be a quite interesting option, mainly because they are available in large amounts and at low costs (Kumar et al., 2000). Moreover, the process has other advantages such as no need for any pretreatment of material, high efficiency and the possibility of recovery of metal adsorbed (Reddy et al., 2010).

Considering the features and advantages attributed to the biological adsorption process, it is fundamental to make attempts to find new technically and economically feasible adsorbent materials (Santos et al., 2010b).

Thus, some biological adsorbents, or biosorbents, such as sugarcane bagasse in the removal of Pb (Santos et al., 2010b) and Cr (Santos et al., 2010a), mussel shells (Peña-Rodríguez et al., 2010) and dry biomass of *Eichornia crassipes* (Gonçalves Jr . et al., 2009) have been explored, regard to the removal of heavy metals in solution.

Considering various species of plants tested around the world, some of them showed a great ability to clarify natural waters containing impurities. One of these species is *Moringa oleifera* Lam., belonging to Moringaceae (Katayon et al., 2006), which, in addition to the clarifying property, has other attractive features, such as high nutritional value and an oil content, between 27 and 40% (Jahn, 1989). This crop emerged in certain regions as an alternative and a good choice for biodiesel production, mainly by adapting to various climatic conditions, having a good tolerance to drought and high availability throughout the year (Sharma et al., 2006).

Most studies related to metals adsorption using different adsorbent materials has been conducted only in monoelementary solutions. However, it is well known that many metals are found simultaneously in effluents, so it is necessary to make studies involving two or more metals in aqueous solutions (Srivastava et al., 2008), Considering the advantages attributed to moringa, the present work aimed to study the reuse of the byproduct from its seeds for the adsorption and removal of heavy metals Cd, Pb and Cr, present in contaminated solutions.

MATERIAL AND METHODS

The experiment was carried out at the Laboratory of Environmental and Instrumental Chemistry, Center of Agricultural Sciences, West Paraná State University, *Campus* Marechal Cândido Rondon, PR, Brazil.

Moringa seeds obtained at Uberlândia, MG, Brazil, were crushed and dried in an oven at 60 °C for 36 h. Subsequently, the seed oil was extracted through a Soxhlet system (IUPAC, 1988) resulting in the byproduct, which was dried again in the oven at 60 °C for 24 h, for complete evaporation of n-hexane used for the oil extraction.

A fraction of the moringa byproduct was submitted to nitropercloric digestion (AOAC, 2005), for determining the concentrations of Cd, Pb and Cr by atomic absorption spectrometry - flame (FAAS) (GBG 932 AA) (Welz & Sperling, 1999).

The procedure for determining the pH_{PCZ} consisted of performing a mixture of 50 mg of the adsorbent in 50 mL aqueous KCl at two concentrations (0.05 and 0.5 mol L^{-1}), with initial pH values ranging from 2.0 to 10.0, which were buffered with solutions of HCl and NaOH (0.01 mol L^{-1}). After the contact for 24 h, the final pH values were obtained and plotted on a graph correlating the variation of initial and final pH, with pH_{PZC} corresponding to the range in which the final pH is kept constant (regardless of the initial pH), i.e., the adsorbent surface behaves as a buffer (Mimura et al., 2010).

Aqueous solutions (1000 mL) fortified with Cd, Pb and Cr were prepared and obtained from an aliquot of the stock standard solution (100 µg mL^{-1}).

The tests ranging the mass were carried out to verify the ideal adsorption conditions, using increasing amounts of adsorbent material (0.0 to 0.900 g) at two pH conditions (5.0 and 7.0), adjusted and buffered with standard solutions of HCl and NaOH (0.100 mol L^{-1}).

In Erlenmeyer flask (125 mL) containing the adsorbent material were added 50 mL of aqueous solution enriched with heavy metals in the following concentrations: Cd (0.05 mg mL^{-1}), Pb (0.10 mg mL^{-1}), Cr (0.50 mg m^{-1}). The concentration chosen for each metal studied in this stage was 10 times the maximum allowed value in accordance with Ordinance N°. 518 from the Brazilian Health Ministry, which establishes procedures and responsibilities relating to control and surveillance of water quality for human drinking and its potability standards (Brasil, 2004).

The flasks were shaken for 90 min at constant temperature and stirring (25 °C and 200 rpm) in thermostat water bath, and then 10 mL aliquots were centrifuged for 5 min at 3000 RPM, in order to separate the adsorbent from the aqueous solution, and subsequently to determine the concentrations of Cd, Pb and Cr in the solution by FAAS (Welz & Sperling, 1999).

The isotherms were obtained based on the optimum conditions established previously in the mass and pH tests. Therefore, 0.300 g of moringa seeds byproduct were added to Erlenmeyer flasks (125 mL) with a solution enriched with increasing concentrations of the toxic heavy metals studied. For this, an adjusted and buffered solution at pH 5.0, was used containing the following metal concentrations: Cd (0.20 µg mL^{-1}), Pb (0.40 µg mL^{-1}), Cr (2.0 µg mL^{-1}), values that correspond to 40 times the maximum allowed value by the Ordinance n. 518 MS (Brasil, 2004).

The flasks were shaken at 200 RPM in water bath maintained at 25 °C for 90 min. Subsequently, aliquots of this solution were centrifuged for 5 min at 3000 RPM, to separate the adsorbent from the aqueous solution. Then, the content of metal in the solutions by FAAS (Welz & Sperling, 1999) was determined using the Eq. 1:

$$Q_{eq} = \frac{\left(C_0 - C_{eq}\right)}{m} V \qquad (1)$$

where:

Q_{eq} - amount of metal adsorbed, mg g^{-1}

m - mass of adsorbent used, g

C_0 - initial concentration of metal in solution, mg L^{-1}

C_{eq} - metal concentration at equilibrium in the solution, mg L^{-1}

V - volume of solution used, L

The percentage of metal removal (R%) from solution was calculated according to Eq. 2:

$$\%R = 100 - \left(\frac{C_{eq}}{C_0} \times 100\right) \qquad (2)$$

where:

%R - metal removal (%) from solution by the seed byproduct of *Moringa oleifera* Lam.

C_{eq} - metal concentration at equilibrium in the solution, mg L^{-1}

C_0 - initial metal concentration in the solution, mg L^{-1}

The linearization of isotherms was carried out using the mathematical models of Langmuir (Eq. 3) and Freundlich (Eq. 4):

$$\frac{C_{eq}}{Q_{eq}} = \frac{1}{Q_m b} + \frac{C_{eq}}{Q_m} \qquad (3)$$

where:

C_{eq} - metal concentration at equilibrium in the solution, mg L^{-1}

Q_{eq} - amount adsorbed at the equilibrium per adsorbent unit mass, mg g^{-1}

Q_m - maximum adsorption capacity, mg g^{-1}

b - parameter related to strength of the adsorbate-adsorbent interaction

$$\log Q_{eq} = \log K_f + \frac{1}{n} \log C_{eq} \qquad (4)$$

where:

C_{eq} - metal concentration at equilibrium in solution, mg L^{-1}

Q_{eq} - amount adsorbed at equilibrium per adsorbent unit mass, mg g^{-1}

K_f and n - related to the adsorption capacity and the heterogeneity of the solid, respectively

Results and discussion

The results of determination of toxic heavy metals in the moringa byproduct were 1.00 µg Cd g^{-1}, 3.93 µg Pb g^{-1} and 0.37 µg Cr g^{-1}.

The presence of from soil heavy metals in plants and seeds is due to absorption from soil or absorption via the application of pesticides or fertilizers, since the fertilizers used to supply micronutrients have toxic heavy metals such as Cd, Pb and Cr in their composition, in addition to the desirable elements (Gonçalves Jr. & Pessoa, 2002). These heavy metals may come from indiscriminate disposal of industrial waste, which contain micronutrients and are sold as fertilizers (Gonçalves Jr. et al., 2000).

Thus, knowing the possibility of inputs application in the moringa crop, the presence of these elements in the byproduct can be justified by the use of these practices.

In order to understand the adsorption mechanism, it is necessary to determine the zero charge point (pH_{PZC}) of the adsorbent (Wang et al., 2008). The results obtained in KCl solutions indicated that the zero charge point for moringa is about pH 4.4 (Figure 1). Thus, the adsorption of cations in case of heavy metals is favored at pH values above the pH_{PZC} (Tagliaferro et al., 2011).

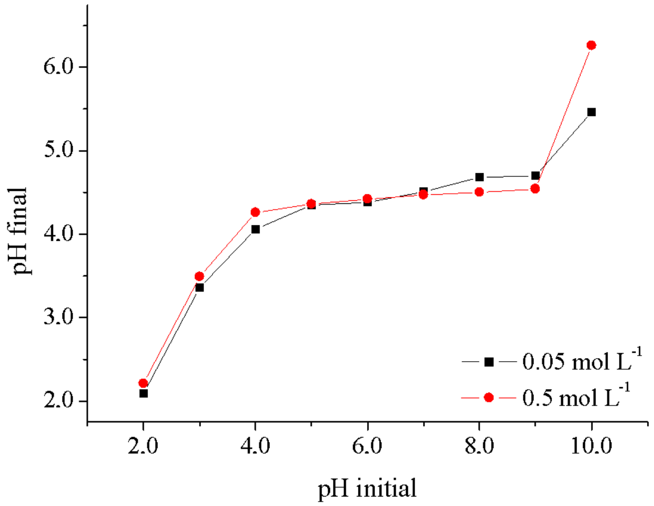

Figure 1. Zero charge point (pH_{PCZ}) of *Moringa oleifera* Lam

The pH chosen was pH 5.0 for solutions in subsequent tests, because, in addition to the pH_{PZC} test, it was also possible to prove in the pH test that fortified solutions at pH 5.0 needed lower mass to achieve a similar efficiency to that obtained under conditions of pH 7.0. One important factor is that lower pH values keep the metal soluble and favor its mobility (Fetter, 1993), confirming the data reported by Sud et al. (2008), whose metals as Cd and Pb the optimum pH values for adsorption using agricultural residues are that with acidic character.

The metal removal efficiency increased with the mass increase, a fact that can be attributed to the increased surface area for adsorption and to the availability of active adsorption sites. However, an excessive increase in the adsorbent amount may cause a reduction in the removal, may be due to the formation of aggregates during sorption, as described by Ekmekyapar et al. (2006). Thus, the mass chosen for the procedure was 0.300 g.

The adsorption isotherms of the studied metals on the moringa byproduct were based on the best results obtained in the tests: 0.300 g of adsorbent and solution at pH 5.0 (Figure 2).

The mean percentage of removal from solution by the adsorbent was 80.2% for Cd, 69.2% for Pb, and 86.7% for Cr. After the isotherms construction, the linearization was performed according to the mathematical models of Langmuir and Freundlich, using the aforementioned Eqs. 3 and 4, respectively.

Table 1 shows the parameters obtained and their correlation coefficients for linear fit of adsorption data according to Langmuir and Freundlich models.

The experimental data of Cd adsorption on the moringa byproduct were similar in both the models used, since the difference between determination coefficients (R^2) was negligible, indicating a possible existence of more than one type of adsorption site interacting with the metal. For Pb, the model that best fitted was Freundlich, indicating that the adsorption occurred in multiple layers (Tarley & Arruda, 2003). Linearization data showed the best fit by Langmuir model for Cr, which shows higher monolayer adsorption.

Values of Freundlich constants (K_f) for Cd, Pb and Cr on the adsorbent ranged from 0.187 to 9.580 (mg g^{-1}) and followed the order: Cr > Pb > Cd. This adsorption sequence can be associated with the characteristic of each metal and the form of interaction with the adsorbent.

Despite Pb and Cr have higher K_f values (adsorption capacity) compared to Cd, these elements showed lower reactivity (n) over it (Cd > Pb > Cr). The parameter "n" indicates the reactivity of the adsorbent's active sites, in the linearization by Freundlich.

Thus, analyzing this parameter shown in Table 1, it can be seen that the processes of Cd adsorption on the adsorbent are shown favorable. According to Sodré et al. (2001), values above 1 for this parameter are a strong indication of the presence of highly energetic sites, suggesting that they were the first to be occupied by the metal, resulting in high energy interaction and high reactivity.

In the Langmuir linearization, it was verified that Cr had a higher adsorption capacity (Q_m) compared to Cd and Pb; however, comparing the parameter 'b', Cd showed the highest binding energy with the adsorbent.

According to Sharma et al. (2006), the aqueous solution containing the seeds byproduct of *Moringa oleifera* Lam. is a heterogeneous mixture containing various functional groups, especially low molecular weight amino acids. These amino acids can be considered an active group of binding agents,

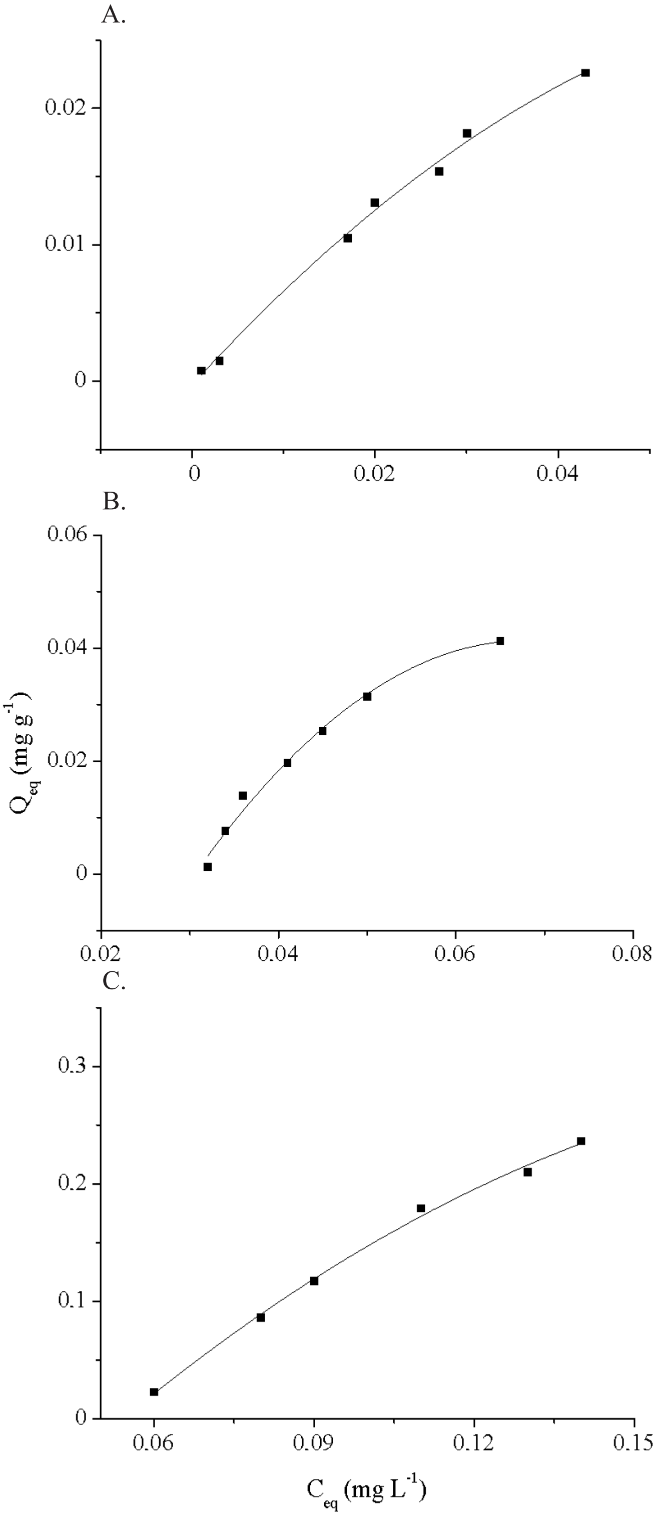

Figure 2. Adsorption isotherms of Cd (A), Pb (B) and Cr (C) on the *Moringa oleifera* Lam. byproduct

Table 1. Parameters of the equilibrium isotherm of Langmuir and Freundlich models for Cd, Pb and Cr adsorption on the moringa byproduct

Metal	Langmuir model			Freundlich model		
	q_m (mg g^{-1})	b (L mg^{-1})	R^2	K_f (mg g^{-1})	n	R^2
Cd	0.02	1443.00	0.98	0.19	1.46	0.98
Pb	0.01	-30.77	0.89	8.98	0.57	0.95
Cr	0.38	-20.45	0.99	9.58	0.54	0.98

acting even at low concentrations due to the ability to interact with metal ions increasing their sorption.

In multielementar solutions, there may be a decrease in the adsorption ability of a specific metal, compared to that of the same metal in monoelementar solutions. This decrease can be attributed to fewer active sites available, i.e., binding sites competitively are divided among metals present in the solution (Sharma et al., 2007), which was observed by the same author in studies using seeds of *Moringa oleifera* Lam. in a ternary solution containing Cd, Cr and Ni, whose adsorption values decreased by about 10 to 20% compared to the adsorption using a monoelementar solution.

Some factors related to electronegativity, hydrolysis constant and ionic radius can favor the adsorption of a metal species over another (Mimura et al., 2010). Regarding the electronegativity, there is the following selectivity order: Pb > Cr > Cd, since the higher the electronegativity, the greater the affinity for sorption (Sag et al., 2002). Regarding the ionic radius, Pb would be the most adsorbed, followed by Cd and Cr. Pb ions have the smallest radius compared to the other metals studied, thus, it has greater accessibility to the surface and pores of the adsorbent, resulting in higher adsorption capacity (Srivastava et al., 2008).

However, in the present study it was observed that Cr ion was preferentially adsorbed over other metal species, this fact may be related to its higher positive charge and ease to form hydrolyzed species, which has a significant effect on the adsorption. The trend to undergo hydrolysis has been observed as the most important factor in adsorption processes involving several metal ions simultaneously (Lesmana et al., 2009). The element with the least ability to undergo hydrolysis is Cd, followed by Pb and Cr. However, the results showed that Cd was more adsorbed than Pb. This can be explained by the Cd larger reactivity (n) of the active sites of the adsorbent, as shown in Table 1.

In addition to the aforementioned properties, the concentration factor can also be taken into account, since the metals were added to the solution at different concentrations and therefore an order of preference cannot be established regarding adsorption selectivity, as the Cr was added to the solution in excess, compared to the other metals.

CONCLUSIONS

1. The adsorption process occurred more efficiently at pH 5.0, its use being feasible as an adsorbent in natural waters that typically have a pH between 5.0 and 5.5.

2. The experimental data of cadmium adsorption on the moringa byproduct were similar in both the models used, indicating a possible existence of more than one type of adsorption site interacting with the metal. Freundlich was the model that best fitted for lead. For chromium, data linearization showed a best fit by the Langmuir model.

3. The moringa byproduct can be considered an alternative for remediation of waters contaminated with toxic heavy metals cadmium, lead and chromium, being a low cost option which requires no previous treatment.

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