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Sorption of heavy metals from aqueous solution by dehydrated powders of aquatic plants

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Abstract Ruppia maritima and Echinodorus amazonicus were prepared in a dehydrated powder form. The characteristics and mechanisms of adsorption of heavy metals were studied under various pH values, reaction times, and heavy metal ion concentrations. The results showed that under different pH and reaction time conditions, heavy metal adsorption was lead > cadmium > zinc > copper. The adsorption of lead increased linearly with the lead concentration. For cadmium, zinc and copper, the adsorption was saturated when metal ion concentration exceeded 200 mg/L. When a Freundlich model was applied, R^2 values for the heavy metal adsorption by the aquatic plants mostly exceeded 0.9. The adsorption of heavy metal ions by these two aquatic plant powders was better explained by the Lagergren second-order equation than the first-order equation. From the Fourier Transform Infrared spectra, there was an adsorption peak at 2,115 cm⁻¹ for *R. mari*tima. The peak shape did not change with metal affiliation except there was a shift of peak wavelength before adsorption. The results indicate that the mechanism of heavy metal adsorption by the two species is not simply on the mono-molecular layer level, and that intra-particulate

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Department of Mathematics, Science, Social Science and Technology, Hong Kong Institute of Education, Hong Kong SAR, China dispersal is the dominant process. Heavy metal pollution does not affect the basic chemical components, and major substances involved in heavy metal adsorption including carbohydrates, cell wall pectin, and protein functional groups.

Keywords Adsorption kinetics · *Echinodorus amazonicus* · Fourier Transform infrared spectra · Langmuir and Freundlich isotherms · *Ruppia maritime*

Introduction

Heavy metal pollution of water is an environmental problem urgently needing to be solved. The sources of aquatic heavy metal pollution are broad; including industry, agriculture, and even sewage from domestic sources can cause aquatic heavy metal pollution (Du et al. 2011; Ikem and Adisa 2011; Kansanen and Venetvaara 1991; Reza and Singh 2010). Heavy metals can be absorbed by aquatic organisms but cannot be degraded and as a result they accumulate through food chains and cause long-term adverse effects at a range of trophic levels (Ahmad et al. 2010; Dizadji et al. 2011; Göbel et al. 2007; Martins et al. 2004; Zvinowanda et al. 2009).

Chemical deposition, ion exchange, membrane filtration, reverse osmosis and adsorption are some of the methods used to control and manage aquatic heavy metal pollution (Pan et al. 2005). Adsorption is the most common and widely used method. Activated carbon is a common adsorbent; however, the cost is enormous, and therefore cheaper, more accessible, environmentally friendly, and more efficient adsorbent materials are being studied (Ng et al. 2003). Seaweed (Gupta and Rastogi 2008), mushroom harvest residues (Saeed et al. 2005), pine bark, pine needles and leaves (Vazque et al. 2007), modified chitosan,



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cassava, and loofah sponge (Saeed et al. 2005), and saw dust (Yu et al. 2001; Vinodhini and Das 2010) have been used as potential adsorbents in research studies.

Submerged plants have large surface areas, and their living bodies have good performance on heavy metal absorption (Ho and Mckay 2000; Miretzky et al. 2006; Ramiro and Rui 2002). It is also possible to use adsorbent plant material in a dehvdrated powder form, but studies and reports on their heavy metal adsorption properties and the impacts of environmental factors on adsorption rate are rare. In this study, two species of aquatic plants were prepared as dehydrated powders and their adsorption characteristics of copper (Cu), cadmium (Cd), lead (Pb), and zinc (Zn), and the influence of environmental factors (initial pH value, reaction time, initial heavy metal ions concentration, etc.) that affect adsorption were compared. The roles of different functional groups in adsorption were described, and they would be valuable references for choosing and using heavy metal adsorbents. The study was done at the environment pollution and toxicity laboratory in Guangzhou of China from November 2010 to March 2011.

Materials and methods

Aquatic plants

Thriving submerged plants Ruppia maritima and Echinodorus amazonicu were collected from greenhouse and brought back to the laboratory. The plants were washed with tap water and de-ionized water, then dried at 80 °C to a constant weight, grounded into powder form, sieved trough a 2-mm mesh, and stored in polyethylene plastic bottles for further use.

Solutions

5 % nitric acid was used to diluted CuSO₄·5H₂O, CdSO₄·8H₂O, Pb(NO3)2 and ZnSO₄·7H₂O to prepare 1,000 mg L⁻¹ Cu²⁺, Cd²⁺, Pb²⁺and Zn²⁺, respectively. The stock was directly diluted to the needed ion concentration in experiments. 1 mol L⁻¹ HNO₃ and 1 mol L⁻¹ NaOH solution were made and used to adjust the pH value. All glassware were immersed in HNO₃ overnight and then rinsed with deionized water before use.

Determination of optimum aquatic plant powder

The two aquatic plant powder in the range 1-40 g/L were added to different flasks containing 100 mg/L from each metal solution to investigate the optimum aquatic plant powder for heavy metal biosorption (Ibrahim 2011). The



maximum biosorption (98 %) of the metal ions was reached when the aquatic plant powder of solutions approaching up to 15 g/L adjusted to pH 6 within 2 h. Therefore, the optimum quantity of aquatic powder was selected as 15 g/L for further experiments.

Optimum pH determination

The optimum pH for heavy metal biosorption was investigated by adding 0.75 g with concentration of 15 g/L aquatic powder to 50 mL heavy metal solution with the initial concentration of 100 mg/L adjusted to various pH values (1–6) using 1 mol L^{-1} HNO₃ solution in a conical flask. The flask is placed in 140 rpm oscillation to react for 2 h. After the reaction time was over, the contents of the flask were filtered through a 0.45-µm hybrid fiber membrane, and the filtrate taken for analysis using Perkin-Elmer AAnalyst 700 flame atomic absorption spectrophotometer with deuterium background corrector. For the adsorption treatments, results from three replicate samples were averaged for the analysis with one control sample of metal (without organisms).

Determination of optimum contact time

0.75 g aquatic powder was added to 50 mL metal solution with the initial concentration of 100 mg/L adjusted to pH 6 using 1 mol L^{-1} HNO₃ in a conical flask. Flasks were shaken at 140 rpm in room temperature. Samples were taken at t = 5, 10, 15, 20, 30, 60 and 100 min, filtered through a 0.45-µm hybrid fiber membrane and analyzed by AAS. Results from three replicate samples and one control sample were averaged for the analysis.

Determination of heavy metal solution with difference concentrations

3.75 g aquatic powder was added to 50 mL metal solution adjusted to pH 6 using 1 mol L^{-1} HNO₃ in a conical flask. The initial metal concentration was 1, 2, 5, 10, 20, 50, 100, 200 and 500 mg/L; flasks were shaken at 140 rpm in room temperature to react for 2 h. Samples were filtered through a 0.45-µm hybrid fiber membrane and analyzed by atomic absorption spectrophotometer (APHA et al. 1998). Results from three replicate samples and one control sample were averaged for the analysis.

Analysis of functional groups in adsorption and measurement of heavy metals

FT-IR (Fourier transform infrared spectroscopy) was used to analyze the functional groups in adsorption to

determine which heavy metals were found in plant samples. Samples were mixed with a KBr tablet. The absorption peaks within wavelength $450-4,000 \text{ cm}^{-1}$ were recorded. The concentration of heavy metals in clear solution, including copper, cadmium, lead and zinc, were measured by atomic absorption Mapping Spectrometer (TAS-986).

Data analysis

Biological adsorption (q (mg/g)) was assessed according to the following equation:

$$\mathbf{q} = m_{\rm ion}/m_{\rm s} = (c_{\rm i} - c_{\rm f})\mathbf{V}/\mathbf{m} \tag{1}$$

where the rate of heavy metal adsorption $\eta(\%) = (c_i - c_f)/c_i$, m_{ion} is the mass of heavy metal ions(mg), ms is the mass of plant samples(g), c_i is the initial concentration (mg/L), c_f is the reacted mass concentration (mg/L), and V is the reaction solution volume (L).

Langmuir and Freundlich adsorption dynamic models may be used to describe the heavy metal absorption process (Febrianto et al. 2009). Langmuir can be expressed in the following form:

$$c_{\rm e}/q_{\rm e} = 1/({\rm b} q_{\rm max}) + c_{\rm e}/q_{\rm max}$$
 (2)

where c_e is the equilibrium concentration (mg/L), q_e and q_{max} are the equilibrium and maximal adsorption capacity (mg/g), respectively, and b is the equilibrium constant (Gupta and Rastogi 2008).

The Freundlich adsorption dynamic model can be expressed in the following form:

$$\ln q_{\rm e} = \ln \mathbf{K}_{\rm F} + (\ln c_{\rm e})/n, \tag{3}$$

where K_F is the Freundlich Coefficient (mg/g), indicating adsorption capacity, and *n* is the Freundlich index indicating degrees of adsorption (Febrianto et al. 2009; Gupta and Rastogi 2008).

Lagergren's first-order and second-order equations are used to describe the kinetics of heavy metal adsorption (Febrianto et al. 2009; Gupta and Rastogi 2008). The linear form of Lagergren first-order rate equation is as follows:

$$\log\left(q_e - q_t\right) = \operatorname{Log}\,q_e - \mathrm{K}_1, \mathrm{ad} \tag{4}$$

where K_1 , ad is the first-order rate constant (min⁻¹), and q_e and q_t are the equilibrium time and adsorption capacity during time period *t* (mg/g), respectively.

The linear form of Lagergren's second-order equation is

$$t/q_t = 1/(K_2, adq_{e_2}) + t/q_e$$
 (5)

where K_2 , ad is a pseudo-second order equilibrium constant (g/mg/min).

Results and discussion

The impact of different factors on heavy metal adsorption in dehydrated aquatic plants powder under different pH, reaction times and initial concentrations showed a high consistency for individual metals. The order of heavy metal adsorption was Pb > Cd > Zn > Cu. Maximum adsorption occurred at a pH of 2 for Pb and at a pH of 3 for Cd, Zn, and Cu (Fig. 1), with the exception of Pb adsorption by R. maritime, which increased gradually with the reaction time; heavy metal adsorption of the two dehydrated aquatic plant powders reached maximum within the first 5 min for all metals (Fig. 2). Under different initial concentrations, the Pb adsorption of the two dehydrated aquatic plant powders increased linearly with Pb concentration and did not appear to reach saturation even at the maximum concentration examined. Heavy metal adsorption of Cd, Zn, and Cu was saturated when the solution concentration reached certain levels (Fig. 3).

The acidity of solution is a crucial factor affecting heavy metal availability and adsorption. It mainly affects the adsorption sites for heavy metals on the surface of adsorbent and the chemical states of metal ions. When pH is low, the linker on the surface of adsorbent materials will be occupied by hydrated hydrogen ions and

Fig. 1 The effects of pH on heavy metal removal percentage of two plant powders





hinder metal ions getting close due to repulsion. When pH value is increased, the concentration of H^+ decreases, exposing more negatively charged linkers. This facilitates the adsorption of metal ions to the active sites. If the pH value is too high, most of the metal ions in solution will form as hydroxide particles, and adsorption cannot proceed (Niu et al. 1993). In the present experiment, for the two aquatic plants, the Pb(II) adsorption was very low when pH value was less than two. The absorption of Cu(II), Cd(II) and Zn(II) was very low when the pH value was less than three. For the two dehydrated aquatic plant powders, the adsorption of Cu(II), Cd(II), Pb(II), and Zn(II) was decreased significantly when pH was above six, possibly as a result of the dissolved states of the metal ions.

Initial concentration of heavy metals affects the adsorption and adsorption mechanisms (Saeed et al. 2005). Generally, adsorption happens at specific sites when the concentration is low, and the specific sites for adsorption will be saturated with the concentration increase until it gradually reaches the point where exchange of occupied sites is the only process occurring (Saeed et al. 2005). Under each combination of pH, temperature and contact time, the increase in the amount of lead adsorbed was proportional to the weight of dehydrated plant material initially present, and the adsorption of Cd, Cu, and Zn was proportional to the

initial concentrations in solution. When the concentration was increased gradually, the adsorption would slow down until it reached saturation. This might be because of the increase in adsorbence with the increase in functional groups in solution, whereby the growth of the availability of specific adsorption sites enhanced the adsorption rate. The increase in adsorbence increased the number of adsorbent particles and reduced the surface area available for additional adsorbence to occur. In addition, under high concentration of adsorbent, particle interaction might cause desorption of metal ions which bind loosely on the surface of the adsorbent (Sangi et al. 2008) and result in reduction of adsorption capacity.

Adsorption isotherms and adsorption kinetics

Fitting the Langmuir model to explain adsorption rates of Pb, Cd, Zn and Cu by the two plants produced wide R^2 variation (Table 1). Except for Zn Freundlich adsorption R^2 of *E. amazonicus* and Pb of *R. maritime* were 0.8538 and 0.8633, respectively; all the other R^2 were greater than 0.9 (Table 1).

The first-order equation in contrast had low R^2 (<0.5) and showed a poor correlation with the experimental data (data not list). The second-order kinetics equation provided a much better fit to the data than the first-order one (Table 2).

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 Table 1
 Langmuir and Freundlich isotherm constants

	Langmuir			Freundlich		
	$q_{\rm max}$ (mg/g)	b	R^2	K _F	1/ <i>n</i>	R^2
E. ama:	zonicus					
Cu(II)	0.6298	0.0249	0.7574	0.0886	0.9649	0.9562
Cd(II)	0.2402	0.0120	0.6484	1.1308	0.5869	0.9004
Pb(II)	1.6737	1.7356	0.9641	3.9276	0.6426	0.9522
Zn(II)	0.4221	0.0107	0.4973	0.1600	0.8487	0.8538
R. mari	tima					
Cu(II)	0.2283	0.0169	0.6290	0.5336	0.7857	0.9609
Cd(II)	0.1873	0.0162	0.6357	2.4571	0.4850	0.9475
Pb(II)	0.6883	1.6142	0.8885	3.3558	0.7816	0.8633
Zn(II)	0.2194	0.0088	0.4202	1.0527	0.5851	0.9358

Table 2 The pseudo-second-order kinetic constants

	$q_{\rm e} \; ({\rm mg/g})$	K ₂	R^2
E. amazonicus			
Cu(II)	0.8482	1.2822	0.9997
Cd(II)	1.9124	1.8701	1
Pb(II)	2.0771	0.8988	1
Zn(II)	1.3346	7.2954	0.9998
R. maritima			
Cu(II)	1.4612	0.5897	0.9996
Cd(II)	1.5588	2.7043	1
Pb(II)	2.1741	0.2765	0.9990
Zn(II)	1.8148	5.1870	0.9997

The Langmuir and Freundlich equations can be used to explain the heavy metal ions adsorption isotherm of adsorbent (Febrianto et al. 2009; Ho and Mckay 1999; Preetha and Viruthagiri 2005). Langmuir is a mono-layer diffusion adsorption dynamic model, which is used to describe the chemical-based adsorption between bioadsorbent and metal ions (Kim et al. 2004). Assuming the amount of adsorbent is limited and the adsorption sites on the surface are homogeneous, the maximal adsorption capacity should be determined by the degrees of saturation on the surface of the adsorbent. The energy of adsorption remains constant. The Freundlich adsorption isotherm is a nonlinear empirical formula (Chang et al. 2003). Assuming that adsorption is happening on a non-homogeneous surface, with the increase in adsorption capacity, adsorption energy will logarithmically reduce to adsorption equilibrium (Ng et al. 2003; Seader and Henly 2006). For the two aquatic plants, the adsorption isotherm equations of Cu, Cd, Pb and Zn are a better fit to the Freundlich model, which indicates the process of adsorption is not a simple mono-layer adsorption.

Adsorption kinetics studies are mainly used to describe the adsorption rate of adsorbent to the adsorbate. The time for adsorbate to stay on solid-liquid interface is controlled by the rate of adsorption. Lagergren first-order and second-order theories are based on a particle dispersion model. By describing the pollutant adsorption constant, the adsorption mechanisms can be explained (Febrianto et al. 2009). The particle dispersion constant (kid) can be found from equation $M_t = \text{kid } t_{0.5}$ (Gupta and Rastogi 2008). If particle dispersion is the only factor that controls reaction velocity, the graph of M_t against $t_{0.5}$ will be a straight line through the origin, and the slope of a straight line is the particle dispersion rate constant. For the two aquatic plants, the adsorption isotherm equations of Cu, Cd, Pb, and Zn were a better fit with the Lagergren second-order equation, indicating adsorption process followed Lagergren second-order mechanism and adsorption rate was controlled by chemical adsorption (Ibrahim 2011), and the particle dispersion mechanism played a dominant role in the whole adsorption process.

Functional groups of heavy metals absorption only R. maritima had a stronger absorption peak than other plants at 2,115 cm⁻¹ (Fig. 4). The peak shape did not change with metal affiliation except there was a shift of peak wavelength before adsorption. The other adsorption spectra of the two species were similar with significant peaks on approximately 3,300–3,400, 2,930, 1,732, 1,650, and 1,540 cm⁻¹, etc. FTIR spectra showed the functional groups mainly included carboxyl, carbonyl, hydroxyl, amino and other functional groups, as well as double bond, triple bond and so on (Fig. 4).

Plant cells are constructed by polysaccharide, protein, lipid composition, containing carboxyl, carbonyl, hydroxyl, amino and other functional groups which can bind with metal ions (Macfie and Welbourn 2000; Amir et al. 2004). The samples of the two aquatic plants had broad absorption peaks at $3,300-3,400 \text{ cm}^{-1}$, which mainly represented -OH and -NH stretching vibration of carbohydrate, cell wall material and proteins. The absorption peak near 2,930 cm⁻¹ could be -CH asymmetric stretching vibration in CH₃ and CH₂. The combined absorption peak at $1,732 \text{ cm}^{-1}$ was the carbonyl peak, which was a common ester and ketone compound found in membrane lipid and cell wall pectin. In the spectra, there were strong absorption peaks near 1,650 and 1,540 cm⁻¹, which represented amide I band (C=O stretching vibration) and amide II band (NH bending





Fig. 4 The FT-IR spectra

vibration and CN stretching vibration), respectively. The special absorption peak at 1,400–1,440 cm⁻¹ represented the C–O stretching vibration in carboxylate ions (–COO–), which are mainly found in pectin and other ingredients. The absorption peak near 1,440–1,317 cm⁻¹ represented the symmetric bending vibration of CH₃ and CH₂ and the shear-type vibration absorption of CH₃ in proteins and cellulose (Yu et al. 2009). The absorption peak near $1,240 \text{ cm}^{-1}$ which represented the amide III bands, was C-N stretching vibration and N-H bending vibration. There might be a C-O-S stretching vibration peak of sulphuric acid ester, CO stretching vibration of carboxyl or phosphate vibration peak. The strong absorption peak near 1,150 and 1,104 cm^{-1} might be the CO stretching vibration of alcohol hydroxyl, ether or ester based, which came from the main component of cell walls, such as carbohydrates (cellulose, hemicellulose). The acromion near and at 1,064 cm⁻¹ was generated by -OH bending vibration and C-O-C stretching vibration; also P-O-C stretching vibration and the vibration of SiO₂ might contribute to the formation of acromion (Kazuo 2009). There was an absorption peak at 620 cm^{-1} , represented SO_4^{2-} . This weak absorption peak indicated there might be little amount of sulphuric acid ester in the biological materials.

The peak shape of the spectra did not change before or after heavy metal adsorption. The results indicated that heavy metal pollution does not cause significant changes in the basic chemical composition of plants, but the adsorption peak of functional groups that are involved in heavy metals adsorption had varying degrees of displacement, and the functional groups that are not



involved in heavy metal adsorption had only small

degrees of adsorption peak displacement, such as -CH

stretching vibration peak near 2,930 cm⁻¹, symmetric

Conclusion

From the results, we conclude that the mechanism of heavy metal adsorption by the two species is not simply on the mono-molecular layer level and that intra-particulate dispersal is the dominant process. Heavy metal pollution does not affect the basic chemical components of the two plants. In these plants, major substances involved in heavy metal adsorption include carbohydrates, cell wall pectin and protein functional groups such as carboxyl, carbonyl, Hydroxyl, amine and double-bonded, triple-bonded and hydrocarbon chains.

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