Cadmium sorption by *Moringa stenopetala* and *Moringa oleifera* seed powders: Batch, time, temperature, pH and adsorption isotherm studies

L. M. Mataka¹, S. M. I. Sajidu²⁺, W. R. L. Masamba¹,² and J. F. Mwatseteza¹

¹Chemistry Department, Chancellor College, University of Malawi, P. O. Box 280, Zomba, Malawi.  
²Harry Oppenheimer Okavango Research Centre, PBag 285, Maun, Botswana.

Accepted 10 February 2010

There is a widespread recognition that the presence of heavy metals such as cadmium in water is hazardous to the environment and human health and their discharge into surface waters is of great concern world wide. The use of natural coagulants such as *Moringa* for heavy metal removal from water replacing expensive imported synthetic coagulants is particularly appropriate for agro-based developing countries such as Malawi. This study was aimed at investigating the potential of *Moringa stenopetala* and *Moringa oleifera* in the removal of cadmium(ii) ions from water. The potential of *M. stenopetala* for cadmium removal was investigated by means of jar test beakers. With an initial cadmium concentration of 7 mg/l, *M. stenopetala* seed powder, at a dose of 2.59 g/100 ml, reduced the concentration of cadmium by 53.8%. Comparison of removal capacities between *M. stenopetala* and *M. oleifera* indicated that *M. stenopetala* was more effective than *M. oleifera* in removing cadmium from water (p < 0.05). Increasing initial cadmium concentration, agitation time and ionic strength reduced cadmium removal. Equilibrium sorption was attained at pH 5 where maximum cadmium removals were 82.7 and 70.7% using *M. stenopetala* and *M. oleifera* respectively. There was a reduction in cadmium removal between 0 to 60°C for *M. stenopetala* and 0 - 40°C for *M. oleifera* before increasing with subsequent temperature increases. It was also shown that cadmium sorption at 30°C and pH 3 for the *M. oleifera* could best be modelled by the Freundlich isotherm whereas the Langmuir model is slightly better than the Freundlich isotherm in the case of the *M. Stenopetala*. The energies of adsorption from Dubinin-Radushkevich models have indicated that cadmium removal using both powders is based on physisorption. The results indicate that *M. stenopetala* and *M. oleifera* have potential in cadmium remediation of polluted waters.


INTRODUCTION

The United Nations Environment Programme’s (UNEP, 2004) Water Policy and Strategy identifies several water focal areas including fresh water scarcity, land based pollution sources, aquatic biological diversity, resource use and management, and knowledge and technology transfer in integrated water management. The Malawi State of the Environment Report notes that water degradation is a major environmental problem that threatens the health and well being of humans and ecosystems (Malawi Government, 2002). Improper disposal of various types of waste, deforestation, and poor agricultural practices that encourage soil erosion and deposition of sediments into the water bodies were identified as the major causes of water degradation in Malawi. Studies of Malawian urban water bodies and wastewater treatment plants reveal that some water quality parameters including heavy metals exceed the World Health Organization (WHO, 2004) acceptable limits (Matope, 2002; Sajidu et al., 2007). Of particular concern are the high levels of cadmium in some of the

*Corresponding author. E-mail: sajidu@chanco.unima.mw. Tel:+265 1 524 222.
water bodies. Cadmium levels of 0.86 mg/l have been reported in Mudi and Limbo streams (Matope, 2002). A recent water quality study of Blantyre streams (Limbe, Nasoko and Mudi) and wastewater treatment plants (Limbe and Soche) recorded levels of cadmium from 0.002 to 0.015 mg/l with most values exceeding the WHO limit of 0.005 mg/l for drinking water (Sajjdu et al., 2007). Cadmium has no beneficial biological importance in the human body and it is a pollutant of global concern (Ayres et al., 1994). Acute cadmium poisoning symptoms are similar to those of food poisoning. It is associated with kidney disease and linked to hypertension. There is also some evidence that cadmium can cause mutations (Carson et al., 1996). The primary adverse health effects observed are lung cancer and kidney damage. In extreme exposure cases, pulmonary edema may develop and cause death. Severe cadmium-induced renal damage may develop into chronic renal failure at which point some form of dialysis or kidney operation will be needed (Norberg et al., 1992). Also cadmium has the ability to alter the rate of ovarian and placental steroidogenesis, thereby adversely affecting normal reproduction in both humans and animals; therefore, cadmium has been added to the list of acknowledged endocrine disrupting chemicals (EDCs) (Ghedrese et al., 2006).

A wide range of physical and chemical processes are available for the removal of heavy metals including cadmium from water. These methods include chemical coagulation using aluminum and ferric salts (Fetokri and Ogunjufowokan, 2002), and cationic (Evans and Li, 2003); electro-chemical precipitation, ultra filtration, ion exchange and reverse osmosis (Nomnchay and Palansamy, 2005). Biological processes have also been investigated using phytoremediation (Prasad and Freitas, 2003; Lyte et al., 1998). A major drawback with precipitation is sludge production. Ion exchange is considered the best alternative technique; however, it is not economically appealing because of high operational cost. Adsorption using commercial activated carbon (CAC) can remove heavy metals from wastewater, such as cadmium, nickel, chromium and copper. However, CAC remains an expensive material for heavy metal removal. Phytoremediation is time consuming hence it is not appropriate for immediate remediation. Furthermore, the plants accumulate a lot of heavy metals in their system, which hinders their metabolism (Cheng, 2003) and can have a possible effect on the food chain (Todd, 2001). Metal salts (Al2(SO4)3 and FeCl3) can concentrate metal ions in water since at low pH aluminium ions are present in a dissolved form (Nomnchay and Palansamy, 2005).

Recently there has been increased interest in the subject of natural coagulants for treatment of water and wastewater in developing countries (Jahn, 1986; Ndebingsere et al., 1995; Sutherland et al., 1994; Gebremichael, 2004; Igwe et al., 2005). Some agricultural by-products such as coconut fiber, maize husks and cobs, and millet stalks are reported to adsorb heavy metals from aqueous solutions (Igwe et al., 2005). Of major interest are the natural coagulants from tropical plants of the family of Moringaceae, which have reported clarifying properties of turbid water (Jahn, 1981). Moringa oleifera is the most widely distributed, well-known and studied species of the family Moringaceae because of its previous economic importance as a source of the commercially important Ben oil and more recently, as a multipurpose tree for food and lands and a source of water purifying agents for developing countries (Morton, 1991; Goote et al., 1997; Williamson, 1975; Palgrave, 1993; van Wyk and van Wyk, 1997). The water soluble Moringa seed proteins possess coagulating properties.

The active agents in Moringa oleifera extracts responsible for coagulation were suggested as the cationic polypeptides (Jahn, 1981; Ndebingsere et al., 1995). Gassenschmidt et al. (1995) reported the isolation from M. oleifera of a flocculating protein of 80 residues with molecular mass of about 6.5 kDa, isoelectric point above pH 10, high levels of glutamine, arginine and proline with the amino terminus blocked by pyroglutamate, and flocculant capacity comparable to a synthetic polyacrylamide cationic polymer. However, a non-protein coagulant was also reported but not characterised (Okuda et al., 2001). Earlier studies showed that M. oleifera seed powder is effective in cadmium remediation of water (Sajjdu et al., 2005; Sharma et al., 2007).

Although the water clarifying properties of M. stenopetala have not been as extensively studied as those of M. oleifera, Jahn (1986) reported that 100-150 mg/l of M. stenopetala was as effective in water clarification as 200 mg/l of M. oleifera. Our earlier studies have shown that M. stenopetala has the capacity to remove lead from water (Mataka et al., 2006). Furthermore, the studies showed that M. stenopetala is more effective in lead sorption from water than M. oleifera. However, to our knowledge, no work has been reported on the use of M. stenopetala in cadmium remediation of water. The objectives of this study, therefore, were to evaluate the potential of M. stenopetala in removing cadmium from water and compare its effectiveness with that of M. oleifera reported earlier (Sajjdu et al., 2005; Sharma et al., 2007).
MATERIALS AND METHODS

Materials

*M. stenopetala* seeds were obtained from Kenya (Whizpop Products Ltd., Nairobi) in 2005. The seeds were identified by the Forestry Research Institute of Malawi. *M. oleifera* seeds were collected from Chilawawa in southern Malawi in 2004 and 2005. Seeds were dehulled by hand and then ground in a coffee mill (National MX-210PN), until a consistent powder was obtained. All pH measurements were determined using a pH meter (Merckm 744), calibrated with buffers at pH 4 and 7; constant temperature treatments were done in a constant temperature water bath (Bath: Haskie Type 000-9384, Thermocell: Gallenkamp No. 65) and shelling was done using a Griffin shaker.

Methods

Standard cadmium solutions were prepared as described by the American Public Health Association (APHA, 1990) and cadmium was determined using atomic absorption spectroscopy (AAS) (Shimadzu AA-680G V-S) at 283.3 nm with an air-acetylene flame.

Effect of dosage

Varying masses of *Moringa* powders were added to 100 ml (in jar test beakers) of water containing 7 mg/l cadmium(II) and the mixture stirred for 1 h. The mixture was filtered by gravity through Whatman No. 1 filter paper and the metal ion concentrations of the filtrate were determined using AAS.

Effect of stirring time

A 100 ml of 7 mg/l of cadmium(II) aqueous solution was added to 1.0 g of *Moringa* seed powders. The aliquots were stirred and withdrawn at different time intervals, and filtered. The residual concentration was quantified using AAS.

Effects of initial cadmium concentration

Whole seed powder (1.0 g) of the *Moringa* species was added to 100 ml of cadmium(II) aqueous solutions within the concentration range of 0.5 - 15 mg/l. The mixture was stirred for 1 h, filtered through Whatman No.1 filter paper and the residual cadmium(II) ion concentration was quantified using AAS.

Effect of sodium chloride

To each 100 ml solution containing 7 mg/l cadmium(II) ion, 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0 mol dm⁻³ sodium chloride, and 1.5 g of either *M. oleifera* or *M. stenopetala* whole seed powders were added and stirred for 1 h. The mixtures were filtered through Whatman No.1 filter paper. The residual cadmium(II) ion concentration was quantified by AAS.

Effect of pH

Cadmium(II) solutions of different pH were prepared by adjusting the pH of a 100 ml solution of 7 mg/l cadmium ions in deionised water using 20.0 mmol dm⁻³ sodium hydroxide or 20.0 mmol dm⁻³ nitric acid. Portions of 1.5 g of *Moringa* whole seed powders were added to the resulting metal solutions and stirred for 1 h using a magnetic stirrer. The residual metal ion concentration was quantified using AAS.

Effect of temperature

A series of 100 ml aqueous cadmium(II) ion solutions (7 mg/l) were treated with 1.0 g of *Moringa* seed powders within the temperature range of 0 - 100°C. The *Moringa* suspension was immersed in a constant temperature water bath and shaken for 1 h. Treatment at 100°C was done in a stable heating oil. After 1 h the mixture was filtered. The residual cadmium(II) ion concentration of the filtrate was determined by AAS.

Equilibrium sorption studies

Portions of 0.25 g of *M. oleifera* or *M. stenopetala* were weighted into 50 ml centrifuge tubes to which 25.0 ml of cadmium solution with varying initial cadmium(II) ion concentrations between 5 - 15 mg/l were added. The sample tubes were then shaken at a constant speed using the Griffin shaker for 2 h. The suspensions were filtered using Whatman No.1 filter paper. The filtrates were collected in separate clean sample bottles and the metal content was determined using AAS.

RESULTS AND DISCUSSION

Effects of dosage

Figure 1 shows the relationship between dosage and percentage cadmium(II) removal. As the *M. stenopetala* powder dosage increases from 0.5 to 2.5 g/100 ml the cadmium(II) removal percentage increases from about 20% to above 58%. *M. oleifera* dosage of 1.0 g/100 ml results in removal percentage of about 20% which increases to about 61% as the dosage increases to 2.5 g/100 ml. This phenomenon is expected since the metal uptake capacity of the adsorbent increases with the increase in dosage as the number of active sites available for metal increases with increase in the amount of adsorbent. Previous studies have shown that *M. oleifera* is effective in removal of heavy metals including cadmium from water (Sajid et al., 2005; Sharma et al., 2006). Recent studies have also shown that *M. stenopetala* is more effective than *M. oleifera* in lead ion removal (Mateka et al., 2006). This present study indicates that *M. stenopetala* seed powders are also more effective than *M. oleifera* in cadmium removal at 95% confidence level. However, at higher biomass dosages the removal capacities of the two powders are almost similar. Jahn (1986) also observes that *M. stenopetala* is better than *M. oleifera* in clarifying muddy water.

Effect of stirring (agitation) time on cadmium removal

Figure 2 shows the effect of stirring time on cadmium(II) removal. The metal removal was very rapid in the first 30
Figure 1. Effect of dosage of *M. stenopetala* and *M. oleifera* seed powders on removal of cadmium(II) ions at initial cadmium(II) concentration of 7 mg/L at 30°C.

Figure 2. Effects of contact time on cadmium removal using 1.5 g *Moringa* seed powders at initial cadmium(II) concentration of 7 mg/L at 30°C.

Min. Later metal ions get back into solution as the stirring time increased. Similar trend was observed for the biosorption of silver ions to alfalfa biomass (Herrera et al., 2003). This can be ascribed to the formation of less stable complexes, which easily break as the time of stirring increases. The weakness of cadmium-Moringa complexes is enhanced by the fact that cadmium due to its high charge density and small size is strongly hydrolysed by water. This results into competition for cadmium between water and binding sites on the *Moringa*. Hence with the increase in stirring time cadmium(II) ions go back into the aqueous solution. At high agitation time the solution mixture could be assumed to have attained equilibrium.

Effects of initial cadmium(II) concentration on metal removal

Figure 3 shows the relationship between initial cadmium(II) ion concentration and percentage removal at fixed *Moringa* powder dosage of 1.0 g/100 ml. The increase in initial cadmium(II) concentration decreased cadmium percentage removals and increased the amount...
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of cadmium(II) uptake per unit mass of the Moringa powders (mg/g). The removal of cadmium(II) ions decreased from 12.9% (0.65 mg/g) to 6.2% (0.092 mg/g) for M. oleifera and 13.1% (0.85 mg/g) to 7.6 (0.113 mg/g) for M. stenopetala. Further, the results indicated that at several initial concentrations tested, M. stenopetala was more effective than M. oleifera for cadmium ion sorption (p < 0.05). This agrees with results from Figure 1, which shows that M. stenopetala is more effective than M. oleifera. Effect of initial metal ion concentration is an important parameter, which determines the maximum uptake capacity of the chelating biomass (Reddy and Reddy, 2003). For a fixed dose of the biomass the total available binding sites are limited, thereby binding the same amount of metal ions. This results in a decrease in percentage removal of the metal ions as the initial metal ion concentration increases. The enhanced metal uptake with an increase in initial metal ion concentration might be due to the increase in the ratio of initial number of moles of cadmium(II) to the available surface area of the powders. These results agree with those reported in literature on other metal ion-sorption using wild cocoyam and steam activated sulphurised activated carbon prepared from sugarcane bagasse pith (Horstall and Spiit, 2004; Krishnan and Anirudhan, 2003).

Effects of ionic strength on cadmium removal

Generally cadmium(II) ion removal decreased with an increase in ionic strength using both seed powders (Figure 4). There was a decrease from 48.1 - 6.9% for M. stenopetala treatment and from 48.2 - 5.4% for M. oleifera treatment. The decrease in cadmium(II) ion removal with ionic strength suggests that the removal reaction involves formation of outer sphere complexes and therefore the interactions might be electrostatic as observed by Krishnan and Anirudhan (2003) on metal removal using other metal ion-biomaterial systems. At high ionic strength the sodium ions can compete for the binding sites with cadmium(II) ions on the polypeptide hence reducing the metal ion removal.

Effect of initial pH

The percentage removal increased sharply to about 80.0% with increase in pH up to pH 5 and then gradually increased slowly to maximum removals of 93.8 and 88.4% for M. oleifera and M. stenopetala respectively (Figure 5). At the cadmium concentration used the calculated pH for cadmium precipitation as hydroxide was pH 9. However, significant removal occurred below the precipitation pH. Furthermore, the optimum pH range for the cadmium(II) ion removal is pH 5 and above. This shows that the metal removal is dominated by sorption onto the Moringa powders and not precipitation as hydroxides. The optimum cadmium removal pH compares well with Sharma et al. (2006) optimum pH of 6.5 using M. oleifera seed powders. Sajid et al. (2006) reported an optimum pH of 7.8 on cadmium(II) ion removal using water and sodium chloride extracts of M. stenopetala and M. oleifera. The reduced sorption efficiency of cadmium ion removal with a decrease in pH could be attributed to the presence of H⁺ ions in the mixture, which compete with cadmium(II) ions for the binding sites. This conclusion agrees with that reported by Herrera et al. (2003) on silver metal ion sorption using alfalfa biomass, Raji et al. (1997) on heavy metal adsorption using saw dust and Horstall and Spiit (2005)

Figure 3. Effect of initial cadmium(II) concentration on metal removal using 1.0 g Moringa powders at 30°C.
on lead and cadmium ion sorption from aqueous solutions using wild cocoyam biomass. Furthermore, if the pH is lowered far below the isoelectric point, \( pI = 9.6 \) for \textit{M. oleifera} (Ghebremichael et al., 2005), any basic groups such as nitrogen are protonated hence the protein will lose its negative charge and contain only positive charges. The positive charges will repel the positive ions, which make it difficult for the protein to form a complex with the metal ions. This is reflected in Figure 5 where at low pH, the percentage metal removal is also low. The effects of high pH are analogous to those of low pH. The acid groups of the protein are deprotonated hence it obtains a large negative charge, which can interact more favourably with the positive metal ions (Logan, 1996).

**Effect of temperature on metal removal**

The percentage removal for cadmium(II) decreased as the temperature increased in the range of 0 - 40°C (65.5 - 48.0%) for \textit{M. oleifera} and 0 - 60°C (68.3 - 26.7%) for \textit{M. stenopetala} treatment before increasing to 95.1 and 98.8% for \textit{M. oleifera} and \textit{M. stenopetala} respectively at 100°C (Figure 6). Results of temperature dependence were similar to those found in stirring time dependence. The observed initial decrease in cadmium percentage removal with increasing temperature also suggests weak binding interaction between the active sites and the cadmium ions, which supports physisorption. Furthermore, physical adsorption reactions are normally exothermic, hence the extent of adsorption generally increases with a decrease in temperature (Salinas et al., 2000). A similar conclusion was obtained by Izanloo and Nasseri (2005) on cadmium(II) removal using ground corncobs powders and Herrera et al. (2003) on silver(I) ions adsorption by alfalfa biomass. The sorption of cadmium(II) starts to increase at 40°C for \textit{M. oleifera} and at 60°C for \textit{M. stenopetala}. As the temperature is increased, a number of bonds in the polypeptides, which are the suggested sorption agents, are weakened. The
The adsorption isotherms of metal removal

Theory and data evaluation

Three models were used to fit the experimental data: Langmuir model, Freundlich and Dubinin-Radushkevich models. The Langmuir equation was chosen for the estimation of maximum adsorption capacity corresponding to seed powder surface saturation. The linearised form of the equation after rearrangement is given in equation (1):

\[
\frac{C_e}{q_e} = \frac{1}{q_{\text{max}}K_L} + \frac{C_e}{q_{\text{max}}} \quad (1)
\]

Where \( K_L \) (dm\(^3\) g\(^{-1}\)) is a constant related to the adsorption-desorption energy and \( q_{\text{max}} \) is the maximum sorption upon complete monolayer saturation of the powder surface. The experimental data were fitted to equation (1) for linearisation by plotting \( C_e/q_e \) against \( C_e \) (Figure 7). The Freundlich model was chosen to estimate the adsorption intensity of the sorbent towards the powder and the linear form is represented by equation (2):

\[
\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (2)
\]

Where; \( q_e \) (mg/g) is the metal ion uptake per unit weight of Moringa powder; \( C_e \) is the concentration of metal ions in solution at equilibrium (mg/dm\(^3\)); \( K_f \) and \( n \) are the Freundlich constants. The value of \( K_f \) is a measure of the degree of adsorption and \( n \) indicates the affinity of the sorbent towards the powder. For \( 1/n \) less than unity, adsorption is the predominant process taking place otherwise desorption becomes predominant. A plot of \( \ln q_e \) against \( \ln C_e \) in equation (2) yielding a straight line indicates the confirmation of the Freundlich adsorption isotherm. The constants, \( n \) and \( K_f \) can be obtained from the slope and intercept respectively.

The Dubinin-Radushkevich model was used to estimate the energy of adsorption of the sorbent towards the seed powders. The linear form of the equation is represented in (3):

\[
\ln q_e = \ln K_{\text{DRF}} - \beta \varepsilon^2 \quad \text{and} \quad \varepsilon = RT \ln(1 + \frac{1}{C_e}) \quad (3)
\]

Where \( q_e \) (mol/kg) is the amount of metal absorbed at equilibrium, \( K_{\text{DRF}} \) (mol/kg) is adsorption capacity of the adsorbent per unit weight (mol/kg), \( C_e \) (mol/dm\(^3\)) is equilibrium concentration of the sorbent in solution, \( \beta \) (mol\(^2\)-kJ/F) is a constant related to the energy of adsorption. A plot of \( \ln q_e \) versus \( \varepsilon^2 \) is a straight line where \( \beta \) and \( K_{\text{DRF}} \) can be obtained from slope and intercept respectively. The mean energy of adsorption is given by equation (4):

\[
E = \frac{1}{\sqrt{2\beta}} \quad (4)
\]

\( E \) less than 8 kJ/mol means that the sorption process is physical in nature otherwise it is chemical.
For this study the data fitted well to all the sorption models, $R^2 > 0.93$, (Figures 7, 8 and 9). From Table 1 the Langmuir data showed that the maximum adsorption $q_{max}$ and the constant related to adsorption energy $K$ for M. stenopetala were greater than those for M. oleifera. The values of $1/n$ for both powders, from Freundlich plots, were slightly less than unity. This indicated that although adsorption was favoured there was also significant desorption taking place as the reaction occurred. From the Dubinin-Radushkevich parameters the values of energy of adsorption for both powders were less than 8 kJmol⁻¹ (Table 1). This supported our observation that cadmium(II) removal by the two Moringa powders was physical in nature. Generally the adsorption data also supported the observation that M. stenopetala is more effective than M. oleifera, that is, $q_{max}$ and $K$ from Langmuir plots, the adsorption energy, and maximum adsorption capacity from the Dubinin-Radushkevich plots were greater than those of M. oleifera. Looking at the $R^2$ values, the sorption reaction for the M. Oleifera could best be modeled by the Freundlich isotherm ($R^2 = 0.971$ for Freundlich and 0.936 for Langmuir) whereas Langmuir model is slightly better than Freundlich in the case of M. Stenopetala ($R^2 = 0.947$ for Langmuir and 0.946 for Freundlich).
Figure 9. Dubinin-Radushkevich isotherm fit for cadmium(II) sorption data using 1.0 g/100 ml M. oleifera or M. stenopetala at 30°C.

Table 1. Parameters from the data of reaction isotherms for the sorption of cadmium on Moringa seed powders at 30°C.

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Conclusion

In conclusion the study has shown that Moringa stenopetala and Moringa oleifera seed powder can be used for cadmium(II) ion removal from aqueous solution. The percent removal of cadmium(II) is increased with increase in the Moringa powder dosage up to 2.5 g of powder per 100 ml. The equilibrium settling time for the adsorption of cadmium(II) is 2 h. The increase in initial cadmium(II) concentration results in a decrease of percent removal of the cadmium(II). As the ionic strength of the solution increases the cadmium(II) removal percent decreases while increase in pH results in increase of the metal uptake. The data for M. Oleifera fit well with Freundlich isotherm model whereas M. Stenopetala can be modelled by both Freundlich and Langmuir isotherms and the adsorption energy at 30°C is consistent of physisorption.

ACKNOWLEDGEMENTS

The authors acknowledge support from the International Science Program (ISP) through International Programme in Chemical Sciences (IPICS) at the University of Uppsala, Sweden [project code MAW 02] for a grant that provided MSc scholarship for Lyed Matakana to study in the Chemistry Department of the University of Malawi.

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