BIOSORPTION AND RECOVERY OF HEAVY METALS FROM AQUEOUS SOLUTIONS BY *EICHHORNIA CRASSIPES* (WATER HYACINTH) ASH

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Heavy metal's release without treatment poses a significant threat to the environment. Heavy metals are non-biodegradable and persistent. In the present study the ash of water hyacinth (*Eichhornia crassipes*), was used to remove six metals from aqueous solutions through biosorption. Results of batch and column experiments showed excellent adsorption capacity. Removal of lead, chromium, zinc, cadmium, copper, and nickel was 29.83, 1.263, 1.575, 3.323, 2.984 and 1.978 μ gg⁻¹, respectively. The biosorptive capacity was maximum with pH >8.00. Desorption in μ gg⁻¹ of ash for lead, chromium, zinc, cadmium, copper, and nickel was 18.10, 9.99, 11.99, 27.54, 21.09, and 3.71 respectively. Adsorption/desorption of these metals from ash showed the potential of this technology for recovery of metals for further usages. Hydrogen adsorption experiments showed significant storage capacity of water hyacinth ash.

Keywords: Metal sorption; Metal desorption; Water hyacinth; Metallic pollution; Biodesorption

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INTRODUCTION

The discharge of heavy metals into aquatic ecosystems has become a matter of concern over the last few decades. Water bodies are being overwhelmed with waste matter of various types. Among the toxic substances reaching hazardous levels are heavy metals (Regine and Volesky (2000). Heavy metals of concern include lead, chromium, zinc, cadmium, and nickel (Ahalya et al. 2003). Metals are mobilized and carried into the food web as a result of leaching from waste dumps, polluted soils, and water. The metals increase in concentration at every level of the food chain and are passed onto the next higher level, a phenomenon called bio-magnification (Paknikar et al. 2003).

Techniques presently in existence for removal of heavy metals from contaminated waters include reverse osmosis, electrodialysis, ultrafiltration, ion-exchange, chemical precipitation, phytoremediation, etc. However, all these methods have disadvantages such as incomplete metal removal, high reagent and energy requirements, or generation of toxic sludge or other waste products that require careful disposal (Ahalya et al. 2003). In this context, considerable attention has been focused in recent years upon the field of biosorption for the removal of heavy metal ions from aqueous effluents (Alluri et al.

2007). The process of heavy metal removal by biological materials is known as biosorption. Biosorption can be defined as "a non-directed physicochemical interaction that may occur between metal/radionuclide species and biomass" (Ahalya et al. 2003). The biosorption process involves a solid phase (sorbent or biosorbent; usually a biological material) and a liquid phase (solvent, normally water) containing a dissolved species to be sorbed (sorbate, metal ion). Due to higher affinity of the sorbent for the sorbate species, the latter is attracted and bound with different mechanisms. The process continues until equilibrium is established between the amount of solid-bound sorbate species and its portion remaining in the solution.

While there is a preponderance of solute (sorbate) molecules in the solution, there are none in the sorbent particle to start with. This imbalance between the two phases creates a driving force for the solute species. The heavy metals adsorb on the surface of biomass; thus, the biosorbent becomes enriched with metal ions. Mechanisms involved in biosorption can be classified taking into account various criteria based on cell metabolism, they are classified as metabolism-dependent and non-metabolism dependent, while based on location of the sorbate species it can be classified as extra cellular accumulation/precipitation, as cell surface sorption/precipitation, or as intracellular accumulation. The adsorbed ions are transported across the membrane by the same mechanism through which metabolically important ions such as potassium, magnesium, and sodium are conveyed. These mechanisms comprise (i) physical adsorption e.g. electrostatic interaction, which has been demonstrated to be responsible for copper biosorption by bacterium Zooglea ramigera and alga Chorella vulgaris (Ahalya et al. 2003), (ii) ion exchange e.g., biosorption of copper by fungi Ganoderma lucidium and Asperigillus niger (Alluri et al. 2007), or (iii) complexation e.g. biosorption of copper by C. vulgaris and Z. ramigera, which takes place through both adsorption and formation of co-ordinate bonds between metals and amino or carboxyl groups of cell walls (Ahalya et al. 2003).

Pretreatment and immobilization are done to increase the efficiency of the metal uptake. The adsorbed metal is removed by a desorption process, and the biosorbent can be reused for further treatments. While choosing the biomass for metal biosorption, its origin is a major factor to be taken into account. Fast-growing plants that are specifically cultivated for biosorption purposes (e.g. weeds) (Regine and Volesky 2000) can be used as biosorbents. Apart from the microbial sources even agricultural products such as wool, rice, straw, coconut husks, peat moss, exhausted coffee (Dakiky et al. 2002), waste tea (Ahluwalia and Goyal 2005), as well as weeds have been tested for metal biosorption with encouraging results (Regine and Volesky 2000; Shawky et al. 2005). Verma et al. (2008) studied ion exchange during the metal biosorption of Cd, Ni, Cu, Cr, Zn, and Pb by using dried mass of hyacinth for biosorption because of their relatively small size and large surface area. This feature offers a convenient basis for the production of biosorbent particles suitable for sorption process. They contain many polyfunctional metal-binding sites for both cationic and anionic metal complexes. Potential metal cation-binding sites of algal cell components include carboxyl, amine, imidazole, phosphate, sulphate, sulfhydryl, hydroxyl, and chemical functional groups contained in cell proteins and sugars (Alluri et al. 2007).

The regeneration of the biosorbent may be crucially important for keeping the process cost down and in opening the possibility of recovering the metals extracted from the liquid phase. For this purpose it is desirable to desorb the sorbed metals and to regenerate the biosorbent material for another cycle of application. The desorption process should yield the metals in a concentrated form, and it should restore the biosorbent close to the original state for effective reuse with undiminished metal uptake and no physical changes or damages to the biomass. Dilute mineral acids (HCl, H₂SO₄, and HNO₃) have been used for the removal of metals from biomass (Ahalya et al. 2003). For adsorption and desorption, Langmuir isotherm represents the equilibrium distribution of metal ions between the solid and liquid phases (Ahmed and El-Gendy 2008).

The Cr⁶⁺ biosorption onto guava seeds for removal from aqueous solutions was studied by Abdelwahab et al. (2007). It was found that external diffusion to the guava seeds' surfaces and intera-particle diffusion are both involved in the sorption process. Some studies involving live plants of water hyacinth showed that it can absorb or adsorb Zn^{2+} , Cd^{2+} (Karkhanis et al. 2006; Hasan et al. 2007), Hg^{2+} (Skinner et al. 2007), Cr^{6+} , and Cu^{2+} (Karkhanis et al. 2006). The nonliving roots of water hyacinth have the ability to accumulate relatively large amounts of uranium from aquatic solution, depending upon pH, initial uranium concentration, contact time, and weight of the roots (Shawky et al. 2005). Hydrogen adsorption is commonly studied with a Sievert-type apparatus (Checchetto et al. 2004). In the present study, the ash of water hyacinth (Eichhornia *crassipes*) plant, which is an abundantly available agro waste in tropical countries such as in the Indo-pak subcontinent was used for the removal of Ni^{2+} , Zn^{2+} , Cd^{2+} , Pb^{2+} , Cr^{6+} , and Cu^{2+} from aqueous solutions. In present work, we are reporting their removal by adsorption onto the ash of water hyacinth (Eichhornia crassipes). To make these metals reusable, 3M HNO₃ was used to promote desorption from the ash. These plants grow wild in Taxila, district Rawalpindi, Pakistan. The maximum removals from solutions of these metals (when range given was 4.01-29.94 µg/mL) were investigated.

EXPERIMENTAL

Five samples of wildly grown water hyacinth (*Eichhornia crassipes*) plants were collected from various locations. These were washed carefully, air-dried, oven dried by procedure already documented (Badr-Uz-Zaman et al. 2002; Shawky et al. 2005), and burnt at 220° C in an electric muffle furnace to obtain ash. Their normal, total, and average fresh weight, dry weight, weight of ash, and weight of ash as percentage of fresh weight were calculated and recorded. The ash was ground and mixed in order to obtain a composite sample. The pulverized ash had a high surface area. The ash was pretreated with deionized water in order to remove already present soluble metal ions (Alluri *et al.* 2007).

The X-ray diffraction pattern of the ash particles powders was collected with a PANalytical, Netherlands, diffractometer (Model 3040/60 X[!] pert PRO) equipped with a Cu K α radiation source following the procedure given by Niasari et al. (2007). Stock solutions of all metals were prepared by a standard procedure. Different concentrations ranging from 5 to 30 µg of metal mL⁻¹ were prepared as described by others (Hasany et

al. 1997; Shawky et al. 2005). The pH values of stock solutions were noted by pH meter (Orion USA made model SA 720) and kept at constant, acidic levels in order to see the effect of biosorbent under the same set of conditions. The pH of Ni²⁺, Zn²⁺, Cd²⁺, Pb²⁺, Cr⁶⁺, and Cu²⁺ stock solutions was kept at 6.54, 6.49, 1.58, 4.95, 6.49, and 4.77, respectively. For this purpose Na₂CO₃/HNO₃ was used for solution of lead, Na₂CO₃/HCl for Cd²⁺, and Na₂CO₃/H₂SO₄ was used for Ni²⁺, Cr²⁺, and Zn²⁺ (Shawky et al. 2005).

In the batch method (Rauf et al. 1999; Shawky et al. 2005), 10 g ash was used for the adsorption of Cd^{2+} , Pb^{2+} , and Cu^{2+} from solutions. At 25° C, 100 mL solutions were used. The triplicate solutions of Cd^{2+} , Pb^{2+} and Cu^{2+} with 10 g ash after shaking for 30 minutes were left for 24 hours undisturbed. The next day these were filtered. The metal content of leachate/filtrate was measured by atomic absorption spectrophotometry (AAS) with a GBC-932 Plus instrument. The quantity of metal already present in ash was measured by AAS by using the procedure of Ryan et al. (2001).

For desorption studies, 3M HNO₃ solutions were prepared by the procedure described in a previous study (Jeffery et al. 1989). The 100 mL HNO₃ solution in triplicate samples was mixed with metal adsorbent (already metal containing water hyacinth ash). These ash samples were magnetically stirred for 30 minutes and left undisturbed for 24 hours. The solutions were filtered on the next day. The concentration of metal ions was measured by AAS as mentioned above. The percentage recovery and desorption in microgram per gram of ash was computed by the documented procedure (Tan 2007).

A column method was applied for the adsorption and desorption of nickel (Ni²⁺), zinc (Zn²⁺), and chromium (Cr⁶⁺). Samples with 10 gram ash were used in each column prepared with Pyrex glass tubes. The average length of the column was 43 cm, the volume of the column was 36 cm³, and the packing density of the column was $0.414g/cm^3$. The average temperature of the laboratory was 25°C for all experiments. Time of flow of solution through column was 24 hours, and the average volume of metal stock solutions was 100 mL. The quantity of metal already present in ash was measured by AAS, using procedure of Ryan et al. (2001). The concentrations of the metal ions in the known solutions and in the leachate were determined with the atomic absorption spectrophotometer. The quantity of metal adsorbed was denoted in µg of metal in g⁻¹ of ash. The percentage adsorption of metal was also calculated (Hasany et al. 1997; Shawky et al. 2005; Tan 2007). The metal biosorption capacity of ash of water hyacinth plant was calculated (Tan et al. 2007; Ahmed and El-Gendy 2008)

For desorption of metals 3M HNO₃ solutions were used (Ahalya et al. 2003; Alluri et al. 2007). This nitric acid before use and after leaching from metal containing ash was analyzed by AAS. The quantity of these metals in μ g desorbed per gram of ash of water hyacinth and the percentage desorption of metal was also calculated (Shawky et al. 2005). The metal desorption capacity was calculated (Shawky et al. 2005; Tan et al. 2007; Ahmed and El-Gendy 2008). For the column study, 100mL 3M HNO₃ solutions were passed through glass columns, and desorption of nickel (Ni²⁺), zinc (Zn²⁺), and chromium (Cr⁶⁺) was calculated as described above. A sample of ash (0.25g) was taken in a Sievert-type apparatus for the study of hydrogen storage in ash of water hyacinth (Checchetto et al. 2004). The apparatus was evacuated up to 10⁻¹ bar at 300°C. The sample was activated at 250° C at 10^{-1} bar pressure. At different temperatures and pressures the hydrogen adsorption weight percentage was noted, and the results are given in Table 6 and Fig. 3.

RESULTS AND DISCUSSION

In the present work we are reporting Ni²⁺, Zn²⁺, Cd²⁺, Pb²⁺, Cr⁶⁺, and Cu²⁺ adsorption onto the ash of the water hyacinth (*Eichhornia crassipes*) plant, wildly grown in Taxila, Pakistan. The maximum removal from solutions of these metals was investigated. In our experiment the main purpose was to remove metallic pollutants from waste water by using Taxilian water hyacinth ash. Fresh weight, dry weight, moisture content, ash content and crude fibre content for five samples of water hyacinth were assessed. The results show 59.2 ±14.63 g fresh weights, 4.8 ±1.16 g dry weights, 3.6 ±1.0 g of ash, and 90.8 ± 0.40 percentage moisture of fresh weight. Dry plants contain 77 ± 2% ash and 1.4 ±0.48% crude fibres. The concentrations of metals in water hyacinth ash were measured by AAS.

The results of Ni²⁺, Zn²⁺, Cd²⁺, Pb²⁺, Cr⁶⁺, and Cu²⁺ adsorption experiments are summarized in Table 1. It describes metal concentration in $\mu g m L^{-1}$, $\mu g 100 m L^{-1}$ of stock solution and adsorption capacity in $\mu g g^{-1}$ of plant ash. It shows that metal adsorption capacity plant ash was high when 30 μ g ml⁻¹ of stock solution were used. Adsorption was found in the following order for batch method Cu = Pb > Zn, while for the column method it was Zn > Ni > Cr. This ordering may be due to physical adsorption as a result of Van der Waals forces between metal ions and ash particles. Ash contains negatively charged carbon particles, and metal ions are positively charged. Heavy metal's particles generally show higher adsorption capacity compared to lighter metals (Alluri et al. 2007). Matai and Bagchi, (1980) reported that the ash of water hyacinth contains oxides of Na, K, Ca, and Mg. This idea is supported by Fig 1 (XRD spectrum for ash particles). The XRD spectrum shows several sharp peaks of various oxides including Ca, Mg, K, Na, Al, Fe, and Si. These oxides are basic or amphoteric. Upon their reaction with water, they produce hydroxides. The pH of ash is increased due to the formation of these hydroxides. The following insoluble hydroxides are formed: Ni²⁺, Zn²⁺, Cd²⁺, Pb²⁺, Cr⁶⁺, and Cu²⁺, and due to low Ksp (constant for solubility product) these are precipitated. Precipitation or co-precipitation is also cause of adsorption onto ash of these metals.

Based on the spontaneous nature of the sorption process, it is possible that mechanism of sorption may be ion exchange in nature. The most important components in the plant's ash (Fig. 1) are oxides/hydroxides of Ca, Mg, K, Na, Al, Fe, and Si. These underlie phenomena such as adhesion, precipitation, co-precipitation, sequential precipitation, surface precipitation, and adsorption (Vengaris et al. 2001; Gupta and Krishna 2008). Van der Waals forces may be responsible for physical adsorption, while precipitation and ion-exchange may be responsible for chemi-sorption (Alluri et al. 2007). The pretreatment of ash with deionized water and adsorption results have also been verified by recent studies (Zhang et al. 2009). The deionized water may remove soluble ions and create sites for attachment of metal ions.

Table 1. Metal Concentration in μ g mL⁻¹, μ g 100 mL⁻¹ of Given Solutions and Adsorption Capacity in μ g g⁻¹ of Plant Ash

Metal Conc. in µg mL ⁻¹ solution	Metal Conc. in µg 100mL ⁻¹ solution	Lead	Chromium	Zinc	Cadmium	Copper	Nickel
0	0	0	0	0	0	0	0
5	500	4.7	4.01	4.90	4.99	4.95	4.63
10	1000	9.8	9	9.91	9.95	9.93	9.78
15	1500	14.73	13.69	14.91	14.66	14.89	14.70
20	2000	19.7	17.30	19.92	19.34	19.87	19.69
25	2500	24.84	21	24.93	23.95	24.87	24.77
30	3000	29.83	24	29.94	28.41	29.83	29.79

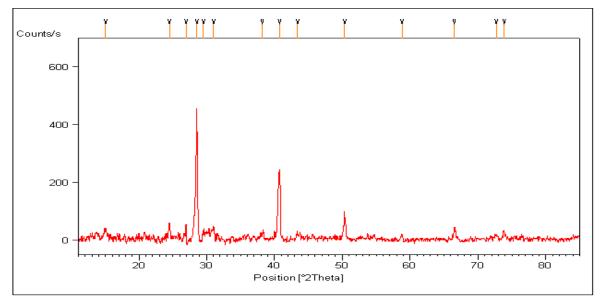


Figure 1. XRD spectrum for ash particles

The bioadsorption factor (BAF) and biodesorption factor (BDF) are very important terms in metal sorption and desorption studies (Tables 2, 3, 4, and 5). We calculated both factors for water hyacinth's ash, as described by Sanchez-Galvan et al. (2008). The BAF increased for Pb²⁺, Zn²⁺, Cu²⁺, and Ni²⁺, decreased for Cd²⁺, and it was variable for Cr⁶⁺. The BDF increased for Cr⁶⁺, Zn²⁺, and Cd²⁺, while it decreased for Pb²⁺, Cu²⁺, and Ni²⁺. The increases in BAF may be due to low K_{sp} , while the decrease in BAF is may be due to high Ksp of oxides and hydroxides. The increase in BDF may be due to high K_{sp} of metal nitrates.

Lead	Chromium	Zinc	Cadmium	Copper	Nickel
0	0	0	0	0	0
0.94	0.802	0.98	0.998	0.9900	0926
0.98	0.900	0.991	0.995	0.9930	0.978
0.982	0.912	0.994	0.977	0.9926	0.983
0.985	0.865	0.996	0.967	0.9935	0.984
0.993	0.840	0.997	0.958	0.9948	0.990
0.994	0.800	0.998	0.947	0.9943	0.993

Table 2. Metal Biosorption Factor of Plant Ash

Figure 2 shows that due to adsorption, pH values were increased. The order of increase in pH was Cd > Cu > Pb > Cr > Ni > Zn. This increase is due to oxides/hydroxides in ash (as shown in Fig. 1 by XRD spectrum) of various elements (Matai and Bagchi 1980). The Cd²⁺, Cu²⁺, Pb²⁺, Cr⁶⁺, Ni²⁺, and Zn²⁺ react with water to produce hydroxides. The hydroxides and oxides are responsible for this increase in adsorption, as described by previous studies (Park et al. 1995; Tamura et al. 1996).

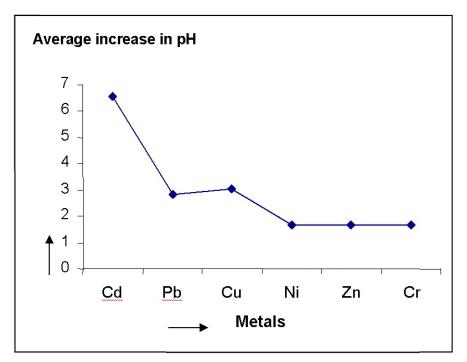


Figure 2. Increase in pH due to adsorption

Tables 3, 4 and 5 show the desorption capacity of metal and BDF. The metal desorption by HNO_3 was low as compared to metal adsorption. This desorption was sufficient to recover these metals for reuse. For the batch method the order of desorption capacity in µg metal g⁻¹ of ash was Cd > Cu > Pb, while for the column method it was Zn > Cr > Ni. These results show that this technique is very useful for the recovery of these metals when present in the range of 5-30 µg mL⁻¹ in metal-polluted water.

Table 3. Adsorbed Pb & Cr in $\mu g g^{-1}$ of Plant Ash, Pb and Cr Desorption in $\mu g g^{-1}$ of Plant Ash and Biodesorption Factor of Pb and Cr

Adsorbed lead onto ash in µgg ⁻¹	Desorbed lead in µgg ⁻¹ of ash	Biodesorption factor of lead	Adsorbed chromium onto ash in µgg ⁻¹	Desorbed chromium in µgg ⁻¹ of ash	Biodesorption factor of chromium
0	0	0	0	0	0
4.7	4.619	0.982	4.01	0.99	0.246
9.8	7.28	0.742	9	2.49	0.276
14.73	10.61	0.720	13.69	3.89	0.284
19.7	12.75	0.647	17.3	5.55	0.320
24.84	15.37	0.618	21	8.57	0.408
29.83	18.10	0.606	24	9.99	0.416

In the process of desorption these metals react with 3M HNO₃ forming nitrates, which are soluble in acidic aqueous media. So these are desorbed and leached from the substrate surfaces. The differences in desorption capacity and BDF are due to variation in K_{sp} of nitrates (Fridenberg, 1974). According to Ahalya et al. (2003) adsorption and desorption can be explained in terms of the following simplifying assumptions:

- Metal ions are chemically adsorbed at a fixed number of well defined sites.
- Each site can hold only one ion.
- All sites are energetically equivalent.
- There is no interaction between the ions.

When the initial metal concentration rises, adsorption increases as long as the binding sites are not saturated. The linearized Langmuir isotherm allows the calculation of adsorption capacities and the Langmuir constants. The concentration of both the metal ions and the biosorbent are a significant factor to be considered for effective biosorption. The concentrations determine the sorbent/sorbate equilibrium of the system. The rate of adsorption is a function of the initial concentration of ions (Ahalya et al. 2005).

Table 4. Adsorbed Zn & Cd in μ g g⁻¹ of Plant Ash, Zn and Cd Desorption in μ g g⁻¹ of Plant Ash and Biodesorption Factor of Zn and Cd

Adsorbed zinc onto ash in µgg ⁻¹	Desorbed zinc in µgg ⁻¹ of ash	Biodesorption factor of zinc	Adsorbed cadmium onto ash in µgg ⁻¹	Desorbed cadmium in µgg ⁻¹ of ash	Biodesorption factor of cadmium
0	0	0	0	0	0.700
4.90	1.19	0.242	4.99	3.99	0.799
9.91	2.59	0.261	9.95	8.97	0.901
14.91	5.12	0.343	14.66	13.66	0.931
19.92	6.99	0.350	19.34	18.41	0.951
24.93	8.99	0.360	23.95	23.03	0.961
29.94	11.99	0.400	28.41	27.54	0.969

Table 5. Adsorbed Cu and Ni in $\mu g g^{-1}$ of Plant Ash, Cu and Ni Desorption in μg per g of Plant Ash and Biodesorption Factor of Cu and Ni

Adsorbed copper onto ash in µgg ⁻¹	Desorbed copper in µgg ⁻¹ of ash	Biodesorption factor of copper	Adsorbed nickel onto ash in µgg ⁻¹	Desorbed nickel in µgg ⁻¹ of ash	Biodesorption factor of nickel
0	0	0	0	0	0
4.95	3.89	0.785	4.63	0.99	0.213
9.93	7.59	0.764	9.78	2.05	0.209
14.89	11.29	0.758	14.7	2.49	0.169
19.87	14.49	0.729	19.69	2.79	0.141
24.87	17.71	0.712	24.77	3.39	0.136
29.83	21.09	0.707	29.79	3.71	0.124

Hydrogen adsorption was studied with a Sievert'z type apparatus, which was calibrated with reference to the ideal gas law, using home-made software. The ash of water hyacinth is an adsorbent for hydrogen. The hydrogen adsorption capacity was found to increase with pressure and temperature as shown in Table 6 and Fig. 3. The trend of increase in H_2 adsorption with changes in temperature and pressure verifies the findings of Checchetto et al. (2004).

Temperature	Pressure in bar	Hydrogen adsorption weight %
27ºC	0.5	0.073%
	1.0	0.082%
	1.5	0.089%
	2.0	0.097%
	2.5	0.097%
50°C	0.5	0.23%
	1.0	0.291%
	1.5	0.312%
	2.0	0.321%
	2.5	0.327%
100°C	0.5	0.271%
	1.0	0.311%
	1.5	0.341%
	2.0	0.393%
	2.5	0.393%
150°C	0.5	0.291%
	1.0	0.334%
	1.5	0.393%
	2.0	0.413%
	2.5	0.417%

Table 6. Hydrogen Adsorption Weight % at Different Temperatures and Pressure

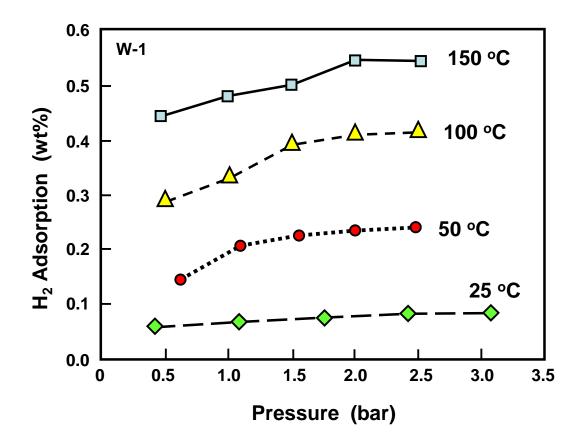


Figure 3. Hydrogen adsorption vs. pressure (At different temperatures)

In view of the foregoing results, the ash of water hyacinth (*Eichhornia crassipes*) can be said to satisfy the criteria for a hyperaccumulator of Pb^{2+} , Zn^{2+} , Cu^{2+} , Cr^{6+} , Zn^{2+} , and Ni^{2+} . The BAF and BDF correspond to the levels shown by Sanchez-Galvan et al. (2008). Hydrogen adsorption experiment showed significant storage capacity. Desorption experiments demonstrated the recovery of Pb^{2+} , Zn^{2+} , Cu^{2+} , Cr^{6+} , Zn^{2+} , and Ni^{2+} from industrial waste water.

CONCLUSIONS

This study showed that the ash of water hyacinth offers several advantages including cost effectiveness, high efficiency, minimization of chemical/biological sludge, and regeneration of biosorbent with possibility of metal recovery. The adsorption capacity of *E. crassipes* ash to metals is more than that of other plant materials. In countries where there is a rush for rapid industrial development coupled with lack of awareness about metal toxicity there is an urgent need for developing an economical and eco-friendly technology that satisfies demands for waste water remediation when other conventional methods fail. Biosorption and desorption by the ash of water hyacinth is

technique can be used for removal and recovery of metal pollutants from water. The hydrogen adsorption by ash shows its potential for use in fuel cells.

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