




Original Research Article

Microwave assisted preparation and applications of bioadsorbents for removal of metal ions from commercial samples

Narendar Bhojak* , Kanhaya Lal, Satya Narayan Jatolia, Uma Rathore

GCRC, P.G. Department of Chemistry, Govt. Dungar College (NAAC 'A' Grade), MGS University, Bikaner 334001, India

ARTICLE INFORMATION

Received: 31 October 2022
Received in revised: 22 December 2022
Accepted: 24 December 2022
Available online: 3 January 2023

DOI: 10.22034/ajgc.2022.4.7

KEYWORDS

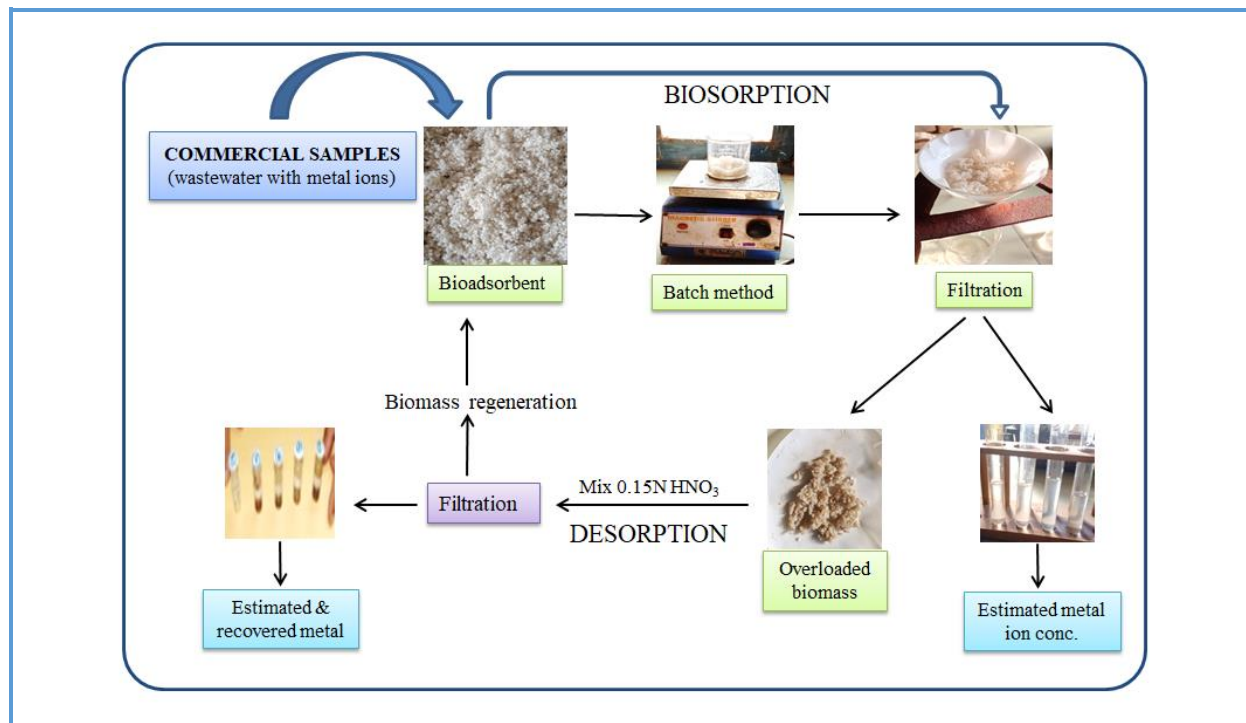
Bioadsorbent
Wastewater
Aerva Javanica
Hazardous Metals

ABSTRACT

Environmental pollution viz water, air, sound, and land pollution, etc. have been a threat to mankind for a long. Water pollution is a major worldwide problem, but in a desert area where already a scarcity of water is there, solid and liquid wastes from different sources make the conditions ugly. Entry of toxic metals in water above a particular concentration is extremely harmful and sometimes lethal, too. In the recent years, the use of bio-adsorbents for the removal of hazardous metal ions from wastewater has been reported, few of these were found suitable whereas many were not, and further improvements and modifications are required to increase the adsorption capacity. In the present paper, the use of microwave radiation for the quick preparation of *Aerva javanica* based bio-adsorbent and its application for the removal of cadmium(II) and lead(II) ions from the aqueous solutions have been described. The *Aerva javanica* Flower adsorbent's metal absorption capability or metal uptake was investigated by batch and column experiments. The experimental stock solutions' initial metal ion concentration, agitation period, temperature, and pH were all examined factors. It investigated how these parameters affected the removal of metal ions. The results of pre-concentration and recovery of metal ions using the column method be 97.8% and 97.2% for cadmium and lead, respectively.

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Graphical Abstract



Introduction

Water pollution is the fouling of water sources by substances that make the water unusable for cooking, drinking, swimming, cleaning, and other activities. Various sources of water pollution by heavy metals have been coal burning in power plants, mining extraction operations, petroleum combustion, metal processing in refineries, textile industries, microelectronics, plastics, etc. The high concentration of metal ions in wastewater is being reduced using different conventional procedures, including precipitation, reduction, adsorption, electro-dialysis, ion exchange, evaporation, and reverse osmosis [1, 2]. The adsorption technique for the removal of heavy metal ions from wastewater has attracted much heed because it is a simple, low-cost, and effective method at low and medium concentrations. Recently researchers are

seeking the synthesis and design of an appropriate Bioadsorbent that has been capable of adsorbing contaminants and has a high adsorption capacity [3].

In recent years, sugarcane waste [4], macromolecule-carbonized rice husk [5], *Chlorella Vulgaris* algal biomass [6], waste plant materials (such as walnut shell, neem wood, pine cone wood, sawdust, almond shell, eucalyptus, etc.) [7], activated carbon obtained from various plant wastes (viz *Mangiferaindica*, acai seed, rubber tire, Plum stones) [8], coffee ground powder [9], and watermelon peels [10] have been used as a bio-adsorbents for removal of hazardous metals (viz Pb, Cd, Hg, Cu, Fe, Mn, Ni, and Cr) from aqueous solution. The goal of the current study is to use inexpensive bio-adsorbents, like the flowering tops of *Aerva javanica*, to remove lead (II) and cadmium (II) from wastewater [11]. *Aerva javanica* is an Amaranthaceae family desert shrub that is also

being used as an anti-diarrheal, anthelmintic, and more recently as an anti-cancer agent [12]. The new green bio-adsorbents are superior and evolved methods have been successfully used for the trace assessment of these metal ions in various samples.

Experimental

Apparatus

A digital pH meter (ECIL model 5651A) and an atomic absorption spectrometer (Shimadzu AA-640-13) were used. In the chromatographic column, a glass column tube of 7 mm and 100 mm i.d. has been used.

Materials and solvents

All chemicals used of AR grade. A stock solution of Pb(II) and Cd(II) ions has been prepared by dissolving their salts in double distilled water and used after standardization. Commercial samples have been collected from industrial areas of Bikaner regions. The Green adsorbents, *Aerva javanica* flowering tops have been obtained from farms in the surrounding area of Bikaner, Rajasthan, and accreditation was done at "Department of Botany, Government Dungar College (NAAC Grade 'A'), Maharaja Ganga Singh University, Bikaner, Rajasthan".

General procedure

Two types of methods (i.e. Batch and Column method) have been used for investigation.

Batch method

Pre-concentration of Pb(II) and Cd(II) ions have been performed by a batch technique at 27 °C. The stock solution was diluted with double distilled water to make various concentrations of metal ions solutions varying from 10-100

ppm. A fixed amount of Green adsorbent (0.1 g) was mixed with 10 mL of various solutions of Pb(II) and Cd(II) ions in a shaker thermostatted to the appropriate temperature and adjusted to the desired shaking speed. The pH of each mixture was maintained between 4-9 (lead 4.0-8.5 and cadmium 5.2-8.7) either by borate buffer or sodium acetate [13]. After a certain time, the green adsorbent is allowed to settle down, the supernatant solution is separated and the concentration of metal ions in the supernatant solution was estimated using an atomic absorption spectrophotometer (AAS) [14].

Column method

In a Column experiment, a glass column with a length of 120 mm and an internal diameter of 8 mm was packed with 0.1 g of green adsorbent to a height of about 14 mm. It was eluted with 6.0 mL of appropriate buffer solution [14] (pH 4 to 9, depending on the optimum pH range of metal ions), afterward, 10 mL (10 ppm) solution of metal ion was maintained for 30 minutes at the same pH, and then allowed to pass through the column at a flow rate of 1.5 mL per minute. The adsorbed metal ions were eluted from the column using 25 mL of 0.15 N HNO₃ and the metal ions were determined using atomic adsorption spectrophotometer (AAS) [15, 16].

Results and Discussion

Effect of operating parameters on adsorption process

Apart parameters have been studied to perusal the effect of initial metal ion concentration, temperature, contact time, pH, and adsorbent dosage on the removal of lead ions using *Aerva javanica* flowering tops adsorbent [17] (Table 1).

Table 1. Parameters studied for batch experimentation

Parametric study	Metal ion concentration (ppm)	Temperature (°C)	Agitation time (min)	pH	Adsorbent dosage (g)
Metal ion concentration	10-100	27	70	6.8	0.1
Temperature	10	20-42	70	6.8	0.1
Agitation time	10	27	40-100	6.8	0.1
pH	10	27	70	4-9	0.1
Adsorbent dosage	10	27	70	6.8	0.02-0.15

Effect of amount of green adsorbent

To explore the effect of the amount of green adsorbent, different amounts of green adsorbent have been investigated, which were prepared by a method previously developed by our group (Figure 1).

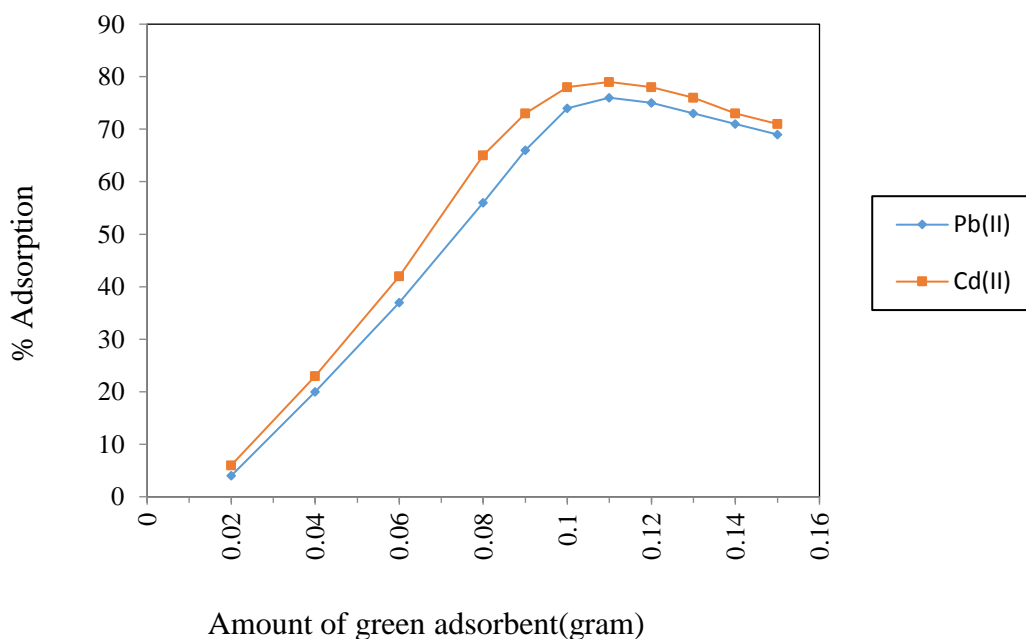
Effect of pH

The conception of metal ions on a column packed with green adsorbent has been studied as a function of pH. Lead and cadmium were retained quantitatively from the aqueous

solution in the pH range of 6.0-8.0 pH and 6.5-8, respectively. The optimum pH selected for further investigation is 6.8 for Pb and 7.5 for Cd [18] (Figure 2).

Optimization of adsorption time

The speed with which the solid phase adsorbs metal ions from the solution and attains a state of equilibrium has of great importance. It was found that the time required to reach equilibrium is about 40 min for Pb and 45 min for Cd, whereas equilibrium was achieved in 5-7 minutes using the microwave radiation.

**Figure 1.** Effect of amount of green adsorbent on the adsorption of metal ions

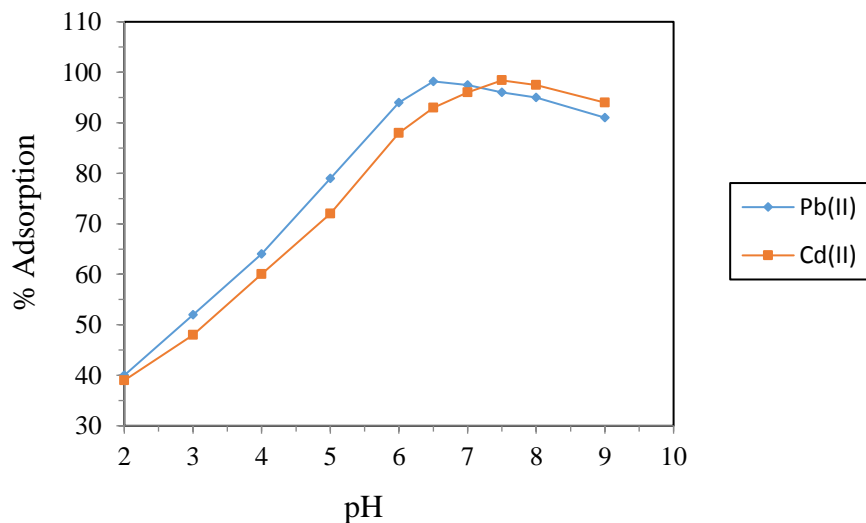


Figure 2. Effect of pH on metal ion adsorption

Adsorption isotherms

The uptake of Cd(II) and Pb (II) by green adsorbents as a function of metal ion concentration in an aqueous solution has been determined using a batch technique. The adsorption isotherm represents a good linear relationship over a relatively wide range of the tested ion concentrations (Figure 3). The distribution coefficient, D , is defined as $D = N1/C$. Here $N1$ is expressed in mmol per gram

and C in mmol per mL. The approximate average value of D (mL per g calculated for the metal in the concentration range 2×10^{-4} M to 2.5×10^{-3} M) has been almost found to be 4.1×10^2 for Pb and 3.9×10^2 for Cd. Plots of adsorption capacity of green adsorbents against the concentration of metal ions had an almost zero intercept (0.002). Regression analysis of the adsorption isotherm plot revealed the correlation was good, $R^2 = 0.995$ [19].

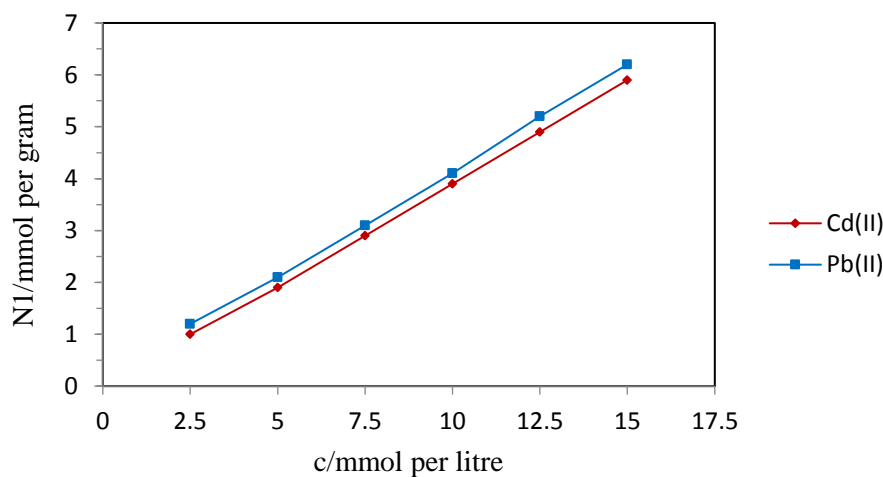


Figure 3. Linear adsorption isotherms

Effect of temperature

The adsorption of a metal ion on an adsorbent is also affected by temperature. The percentage adsorption and distribution coefficient of the complex reaction has been found to increase with an increase in temperature up to 30 °C, and then it remained constant up to 40 °C, followed by a slight decrease.

Effect of flow rate

Adsorption of the metal ions on the green adsorbent column was studied at different solution flow rates. For each metal ion solution (100 mL) containing 50 µg of the ion was passed through a column at a flow rate varying from 1 mL to 4 mL per minute. The adsorption is quantitative and reproducible in the given range. The flow rate, maintained at 2.5 mL per minute, was best suited for better adsorption.

Preconcentration and recovery of metal ions

To study the performance of green adsorbent for pre-concentration of Pb (II) and Cd (II), each metal (40 mg) in different volumes of samples (20-90 mL) was passed through a column at 2.5 mL per min. Elution of the metal ions from the columns resulted in recoveries Pb (II) and Cd(II) was 97.2% and 97.8%, respectively. The uptake of metal ions decreases with increasing sample volume. These results suggest that the use of green adsorbent can effectively concentrate these

metal ions in large quantities from dilute aqueous solutions [20] (Table 2).

Effect of electrolytes

The effect of various electrolytes viz calcium chloride, potassium nitrate, sodium chloride, and magnesium sulfate on the adsorption of metal ions by green adsorbents have been investigated. It was observed that there was no effect of the electrolytes on the adsorption of metal ions by the green bio-adsorbents.

Determination of Lead and cadmium in different commercial samples

100 mL of commercial sample was taken from various sites; metallic contents were separated and dissolved in hydrochloric acid and sulphuric acid successively. After filtration residue was dissolved in nitric acid. 50 mL of the commercial sample was transferred into a 100 mL Erlenmeyer flask. It was digested several times with nitric acid and sulphuric acid. Most of the acid was evaporated and the rest was neutralized with 1% NaOH solution. Finally, the total volume was made with double distilled water. Aliquots of 2 mL from this solution were taken and diluted to 10 mL while adjusting the pH at 7.0 with the borate buffer solution. This solution was passed through a column packed with 0.1 g *Aerva javanica*, and then it was eluted with 0.1N HNO₃ and lead contents were determined using AAS. The recovery was found to be 96-98% [21]. Results are presented in Table 3.

Table 2. Preconcentration and recovery of Metal ions

Metal ions	Conc. (µg/mL)	Vol. of eluent (mL)	Recovery	
			(µg/mL)	%
Pb	10	20	9.72	97.2
Cd	10	20	9.78	97.8

Table 3. Determination of lead and cadmium in commercial sample

Sample	Determination of lead			Determination of cadmium		
	Pb added ($\mu\text{g/mL}$)	Pb det. ($\mu\text{g/mL}$)	% Recovery	Cd added ($\mu\text{g/mL}$)	Cd det. ($\mu\text{g/mL}$)	% Recovery
(A)	-	15	-	-	14.9	-
	10	24.1	96.4	10	24.3	97.59
(B)	-	12	-	-	17.4	-
	10	21.5	97.7	10	26.9	98.17
(C)	-	18	-	-	15.2	-
	10	27.4	97.5	10	24.7	98.01
(D)	-	11	-	-	13.5	-
	10	20.4	97.1	10	22.9	97.44

Conclusion

1. GMBR can be used effectively for the preparation of green adsorbent.
2. The Pb(II) and Cd(II) ions can be effectively removed from various samples using these green adsorbents based on *Aerva javanica*.
3. The solid phase extraction method shows fast method ion-exchange kinetics and high sorption efficiency.
4. The proposed method is widely applicable for the measurement and recovery of trace Pb and Cd in various commercial samples.

Disclosure Statement

No potential conflict of interest was reported by the authors.

Funding

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Authors' contributions

All authors contributed to data analysis, drafting, and revising of the paper and agreed to be responsible for all the aspects of this work.

Orcid

Narendar Bhojak  0000-0002-6404-4219

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How to cite this manuscript Narendar Bhojak, Kanhaya Lal, Satya Narayan Jatolia, Rathore Uma. Microwave assisted preparation and applications of bioadsorbents for removal of metal ions from commercial samples. *Asian Journal of Green Chemistry*, 6(4) 2022, 388-395. DOI: 10.22034/ajgc.2022.4.7