International Journal for Multidisciplinary Research
One Day National Seminar on "Computational Science: The Intersection of Math, Physics, Chemistry and Computer Science"
Organized by Sri Venkateshwara Government Arts & Science College(Autonomous), Palem-509215, Bijinepally, Nagarkurnool Dist., Telangana, India.
E-ISSN: 2582-2160 ● Website: www.ijfmr.com ● Email: editor@ijfmr.com

Application of Column Chromatography for the Separation of Lead (Pb^{2+}) and Cadmium (Cd^{2+}) Ions from their Mixture

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Abstract:

In this study, a low-cost carbonaceous bio-adsorbent was developed from the powdered fruits of Casuarina equisetifolia, an agricultural waste product. The material was chemically modified through treatment with analytical grade nitric acid and sulfuric acid to enhance its adsorption capabilities. Column chromatography experiments demonstrated that the treated bio-adsorbent exhibits significant sorption capacity for the effective separation and removal of lead (Pb^{2+}) and cadmium (Cd^{2+}) ions from their aqueous mixture. The research also explores the potential for recovery of these heavy metal ions and the regeneration of the adsorbent, highlighting its reusability and environmental relevance in wastewater treatment applications.

Keywords: Adsorption, Column Chromatography, Bio-adsorbent, *Casuarina equisetifolia*, Lead (Pb^{2+}), Cadmium (Cd^{2+}), Nitrated Material, Sulphonated Material, Heavy Metal Removal.

I. INTRODUCTION:

Partition techniques encompass distillation, precipitation, ion exchange, solvent extraction, and various chromatographic methods. Among these, chromatography is widely used for the separation and removal of components from mixtures. Ion exchange chromatography, in particular, involves the reversible exchange of ions between a solid, insoluble porous matrix and an aqueous solution brought into contact with it. The ion exchange properties of natural materials such as zeolites and clays have been well understood and studied for over a century. Synthetic ion-exchange resins, which are high molecular weight polymers containing a large number of ionic functional groups, have been developed to enhance this process. Cation exchange resins typically include functional groups such as $–SO_3H^+$ and –COOH, while anion exchange resins carry basic groups like secondary, tertiary, or quaternary amines (e.g., $–N(CH_3)_3^+OH^-$). These resins can effectively separate both organic and inorganic ionic species. Previous studies, such as that by Srivastava, have demonstrated the successful removal of heavy metals

using carbonaceous materials derived from waste fertilizer slurry. Similarly, Balsubramanium reported the use of waste tea leaf ash and fly ash as decolorizing agents for treating dye-contaminated wastewater. The present research focuses on the removal and regeneration of heavy metals, specifically Pb^{2+} and Cd^{2+}, from aqueous mixtures. Initial results using an adsorbent material with a mesh size of 0.63 mm indicate it to be highly effective for the adsorption of both Pb^{2+} and Cd^{2+} ions. Column chromatographic studies utilizing this material as the stationary phase have been conducted, showing promising results for the separation and recovery of these heavy metal ions.

II. MATERIALS AND METHODS

i. Materials and Reagents

The raw material used in this study was *Casuarina equisetifolia* fruits, collected as agricultural waste. All reagents utilized in the chemical treatment and experimental procedures were of analytical reagent (AR) grade, including hydrochloric acid (HCl), nitric acid (HNO_3), and sulfuric acid (H_2SO_4). Column chromatography was employed as the separation technique, and complexometric titration was used for the quantitative analysis of metal ions (Pb^{2+} and Cd^{2+}).

ii. Sample Preparation

Fruits of *Casuarina equisetifolia* were thoroughly washed and sun-dried, followed by oven drying to ensure complete removal of moisture. The dried fruits were then crushed and ground into a fine powder. The powdered material was chemically treated using AR-grade sulfuric acid and nitric acid in a controlled ratio. This acid treatment aimed to activate the surface and introduce functional groups that enhance adsorption capacity.

After chemical treatment, the material was repeatedly washed with distilled water until the washings were neutral, indicating the removal of residual acids. The cleaned material was then dried in a hot air oven at 110°C for several hours to obtain the final bio-adsorbent. This adsorbent was sieved through standard mesh sizes to obtain three distinct particle size fractions:

- **Size-I**: 18 µm mesh
- **Size-II**: 25 µm mesh
- **Size-III**: 63 µm mesh

Each size fraction was collected separately for comparative adsorption and column studies.

iii. Column Preparation

Stock solutions of lead (Pb^{2+}) and cadmium (Cd^{2+}) ions were prepared using their respective nitrate salts, maintaining a concentration of 1 mg/mL. For the column experiments, the downflow method was adopted.

For each trial, 20 g of the Size-III (0.63 mm) bio-adsorbent was soaked in distilled water and stirred for 15 minutes to ensure homogeneity and remove air bubbles. The hydrated material was then packed into a glass chromatographic column of 30 cm length and 30 mm inner diameter. A small plug of glass wool was placed at the base of the column to prevent the loss of adsorbent particles during the experiment.

A mixture of 50 mL aqueous solution containing both Pb^{2+} and Cd^{2+} ions was introduced into the column

at a controlled flow rate of 5 mL/min. The elution process was monitored, and samples were collected periodically for analysis.

iv. Column Separation and Analysis

The breakthrough capacity of the adsorbent was determined by collecting and analyzing successive 50 mL fractions of the eluent. The quantity of metal ions retained by the adsorbent was assessed by continuing the process until no metal ions were detected in the eluent, indicating complete saturation of the adsorbent.

Elution of the adsorbed metal ions was carried out using hydrochloric acid (HCl) and nitric acid (HNO_3) as eluents. The metal ions released during elution were collected and quantified using complexometric titration, employing suitable masking and indicator agents.

The total amount of Pb^{2+} and Cd^{2+} ions adsorbed and subsequently desorbed from the Size-III and Size-IV adsorbents (with particle sizes of 0.63 mm, 0.25 mm, and 0.18 mm respectively) was calculated. Comparative efficiency among the different particle sizes was evaluated based on adsorption capacity and percentage recovery after elution.

III. RESULTS AND DISCUSSION:

Table 1 illustrates the influence of adsorbent particle size on the recovery efficiency of metal ions. It was observed that the **percentage recovery of Pb^{2+} ions increased with increasing particle size** of the carbonized bio-adsorbent derived from *Casuarina equisetifolia* fruits. Specifically, the **highest recovery rate of 82% for Pb^{2+}** was achieved using the **adsorbent with a particle size of 0.63 mm** (Size-III). This suggests a favorable interaction between the larger particle surface and the Pb^{2+} ions, likely due to better porosity and surface area distribution that facilitates adsorption and subsequent elution.

Table 2 presents the percentage removal efficiency of Cd^{2+} ions. The **removal efficiency for Cd^{2+} was found to be 70.97%**, which, although substantial, was lower compared to Pb^{2+}. This difference may be attributed to the comparative ionic radius, charge density, or coordination behavior of Cd^{2+}, which might affect its affinity towards the functional groups present on the treated adsorbent surface.

The findings clearly demonstrate that **the adsorbent material with a 0.63 mm mesh size (Size-III)**, chemically treated using **nitric acid**, is particularly effective for the quantitative removal and recovery of Pb^{2+} and Cd^{2+} ions. Nitric acid, being a strong oxidizing agent, likely enhances the surface functionality of the adsorbent, introducing more active sites that favor metal ion binding.

While these initial results are promising, especially for Pb^{2+}, **further in-depth studies are required** to optimize treatment conditions, improve Cd^{2+} removal efficiency, and assess long-term performance and regeneration cycles. Additional parameters such as contact time, pH, column bed height, and the presence of competing ions should also be investigated to fully understand and enhance the effectiveness of this bio-adsorbent in practical wastewater treatment applications.

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Metal ion	Amount sorted (mg)	Amount recovered by Size-IV (mg)	% Recovery by Size-IV	Mesh Size in mm
Cd^{2+}	25	16	64	**0.18**
Cd^{2+}	25	17	68	**0.25**
Cd^{2+}	25	17.7	70.9	**0.63**
Pb^{2+}	25	18	72	**0.18**
Pb^{2+}	25	19	76	**0.25**
Pb^{2+}	**25**	**20.5**	**82**	**0.63**

Table-1: The % Recovery (Sorption) by S-III for Pb^{2+} and Cd^{2+}

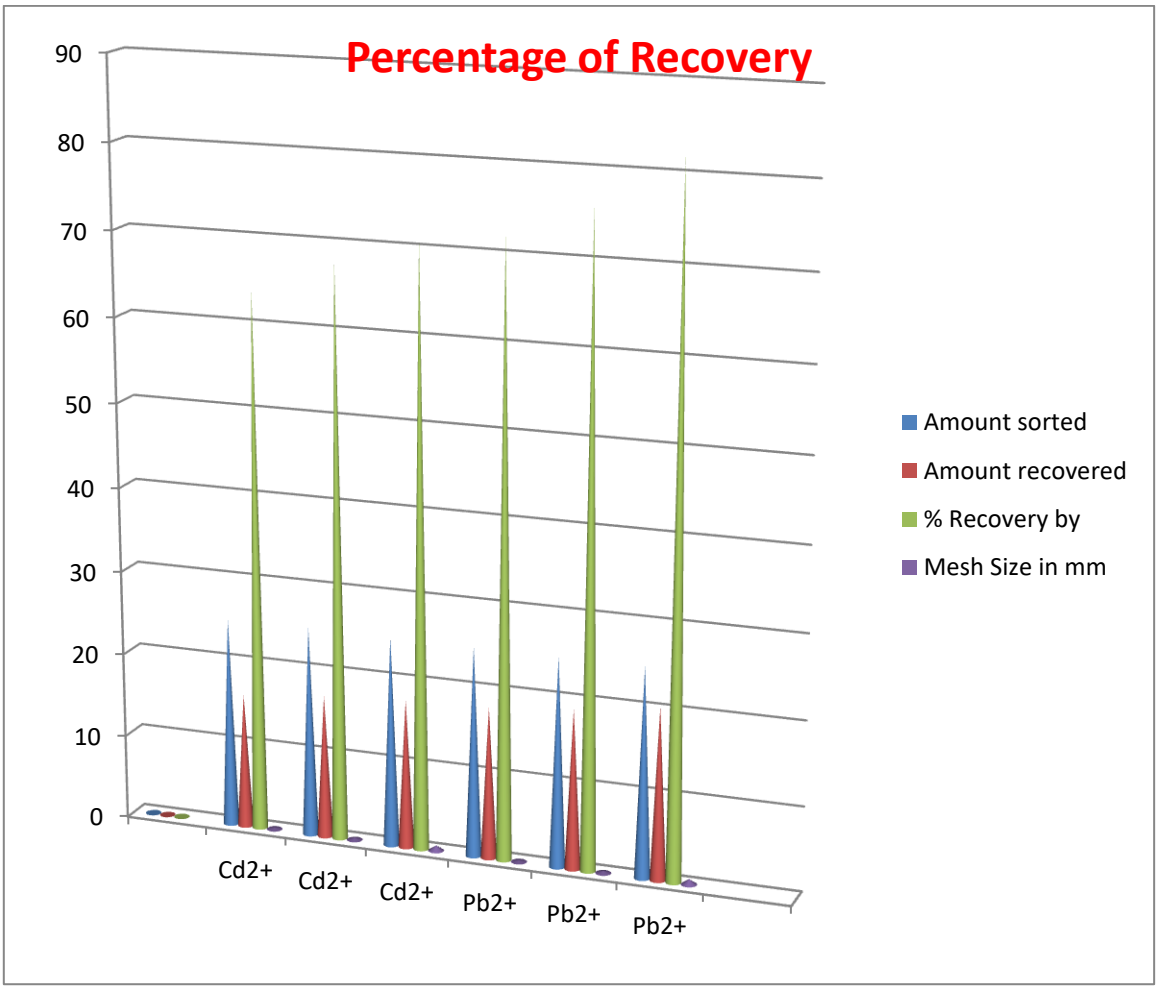


Fig-1: The % Recovery (Sorption) by Size-III forPb^{2+} and Cd^{2+}

IV. CONCLUSION

The present study demonstrates the effective application of a low-cost, eco-friendly carbonaceous bio-adsorbent derived from *Casuarina equisetifolia* fruits for the separation, removal, and recovery of heavy metal ions, specifically Pb^{2+} and Cd^{2+}, using column chromatography. The adsorbent material, chemically modified using nitric and sulfuric acids, exhibited significant adsorption efficiency, with optimal results obtained using a particle size of 0.63 mm (Size-III).

Experimental findings revealed that **Pb^{2+} ions were more effectively removed and recovered** compared to Cd^{2+} ions, achieving **percentage recoveries of 82% and 70.97%**, respectively. This highlights the potential of the treated bio-adsorbent in selective heavy metal ion remediation. The strong oxidizing nature of nitric acid likely contributes to enhancing the surface activity of the adsorbent, thereby improving metal binding affinity.

Overall, this study confirms that agricultural waste materials such as *Casuarina equisetifolia* can be successfully transformed into valuable adsorbents for environmental cleanup. However, to fully establish the practicality of this method for large-scale applications, **further research is recommended**, focusing on regeneration cycles, adsorbent stability, and real wastewater system performance.

V. REFERENCES

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