



# Separation of $Pb^{2+}$ and $Cd^{2+}$ Ion From Their Mixture by Using Column Chromatography Technique.

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**Abstract:** The powdered material prepared from agricultural waste Casuarina equisetifolia fruits has been transformed in to economically cheaper carbonaceous bio-adsorbent by using treatment of analytical grade Nitric acid and Sulphuric acid. Experimentation shows that Sorption capacity is good for removal of heavy metals ion  $Pb^{2+}$  and  $Cd^{2+}$  from their mixture for this adsorbent. Existing research work deals with elimination and recovery of heavy metals ion like  $Pb^{2+}$  and  $Cd^{2+}$  from their aqueous mixture and regeneration of the adsorbent material.

**Keywords:** Adsorption, Column studies, Nitrated material, Sulphonated material and Casuarina equisetifolia fruit.

## I. INTRODUCTION

Partition techniques incorporate distillation, precipitation, ion replace, extraction and a variety of chromatographic techniques. Chromatography is a usually utilized technique for removal of constituents. Exchange of Ion chromatography is a procedure by which ions alleged on a permeable, fundamentally unsolvable hard are switch for ions from a aqueous solution that is fetched into make contact with the solid. The properties of exchange of ion- zeolites & clays have been fine familiar & deliberate for extra than a century. Artificial ion-exchanger resins are with elevated-molecular mass polymers that enclose great amounts of an ionic functional group apiece molecule. A cation exchanger resin contains groups like  $-SO_3H^+$  &  $-COOH$  whereas alkaline functional groups are present in anion exchanger resins like secondary, tertiary or quaternary amine like  $[-N(CH_3)_3^+OH^-]$ . Cation & Anion exchanger resin have the potential of separation of both organic & inorganic ionic species. Adsorption study by Srivastava on removal of heavy metal ions using carbonaceous material obtained from left-over slurry of fertilizer plants<sup>5</sup>. Balsubramanium have stated use of waste tea leaves ash and fly ash as decolourising agents for different dye solution wastes<sup>9</sup>. The current study shows research work on elimination and regeneration of heavy metals like  $Pb^{2+}$  and  $Cd^{2+}$  from their mixture. Results of mesh size 0.63 mm indicated that it is good adsorbent for  $Pb^{2+}$  and  $Cd^{2+}$ . Preliminary experiments on column chromatographic studies using 0.63 mm size adsorbent as a stationary phase materials have been carried out for the separation and recovery of  $Pb^{2+}$  and  $Cd^{2+}$  from their mixture

## II. MATERIALS AND METHODS

### i. Materials and Reagents:

Casuarina equisetifolia fruits, Analytical grade reagent: HCl,  $HNO_3$ ,  $H_2SO_4$  Column Chromatographic, technique, complex-metric titration.

## ii. Sample Preparation:

Fruits of *Casuarina equisetifolia* were dried and converted into powder material, and then with the help of AR. grade Sulphuric acid and Nitric acid simple chemical treatment is given. It is then washed continuously with distilled water till it will be free from acid. This material is dried at 110°C. The dried treated material was allow to pass through sieves of pore size 63 micron mesh, 25 micron mesh, 18 micron mesh size to get different particle sizes Size-I, Size-II and Size-III respectively.

## iii. Column Preparation:

Stock solutions having concentration 1 mg/ml was prepared for  $Pb^{2+}$  and  $Cd^{2+}$  with the help of nitrates of the two metals. Down flow method of column were studied. This experimentation required 20 g adsorbent of different particle sizes of Size-III was added into the distilled water and stirred for 15 minutes and then it is transferred into a glass column having length 30 cm with inner diameter 30 mm. To avoid the loss of adsorbent along with flow of liquid at the bottom of glass column glass wool plug was fitted. Metal ion solution of volume 50 ml having  $Pb^{2+}$  and  $Cd^{2+}$  was introduced in to the column by maintaining flow rate of 5 ml/min.

## iv. Column Separation:

The exhaustive ability was determined by collecting 50 ml portion of the eluent after break through. Same experiment was carried out using Size-III adsorbent. The quantity of metal ions held by the sorbent was discovered. The sorbed metal ion  $Pb^{2+}$  and  $Cd^{2+}$  introduced in the column were collected until the complete metal ion was removed by using eluents HCl and  $HNO_3$ . Complex-metrically the quantity of metal ion extracted from the sorbent after elution was determined. The amount of metal ion held by sorbent and recovered after elution, from columns of Size-III and Size-IV of the different particle sizes (0.18, 0.25 and 0.63 mm) were calculated.

## III. RESULTS AND DISCUSSION:

Table-1 shows that the recovery of the metal ions is get affected by particle size. Percentage recovery increases with increase in particle size; the percentage recovery for  $Pb^{2+}$  is 82 % for carbonised material prepared from fruits of *Casuarina equisetifolia* of particle size 0.63mm. Table-2 shows the percentage removal of  $Cd^{2+}$  is 70.97% indicating less tendency of removal as compared to  $Pb^{2+}$ .

These results indicate that using 0.63mm adsorbent prepared from fruits of *Casuarina equisetifolia* as sorbents removal and recovery of  $Pb^{2+}$  and  $Cd^{2+}$  take place quantitatively. Relatively it was found that sorbent Size-III has good removal and recovery capacity may be this is due to nitric acid is a strong oxidizing agent. Yet further more study is required which would throw light upon the usefulness and effectiveness of these materials for removal and recovery of heavy metal ions.

| Metal ion        | Amount sorbed (mg) | Amount recovered by Size-IV (mg) | % Recovery by Size-IV | Mesh Size in mm |
|------------------|--------------------|----------------------------------|-----------------------|-----------------|
| Pb <sup>2+</sup> | 25                 | 18                               | 72                    | 0.18            |
| Pb <sup>2+</sup> | 25                 | 19                               | 76                    | 0.25            |
| Pb <sup>2+</sup> | 25                 | 20.5                             | 82                    | 0.63            |
| Cd <sup>2+</sup> | 25                 | 16                               | 64                    | 0.18            |
| Cd <sup>2+</sup> | 25                 | 17                               | 68                    | 0.25            |
| Cd <sup>2+</sup> | 25                 | 17.7                             | 70.9                  | 0.63            |

Table-1: The % Recovery (Sorption) by S-III for Pb<sup>2+</sup> and Cd<sup>2+</sup>

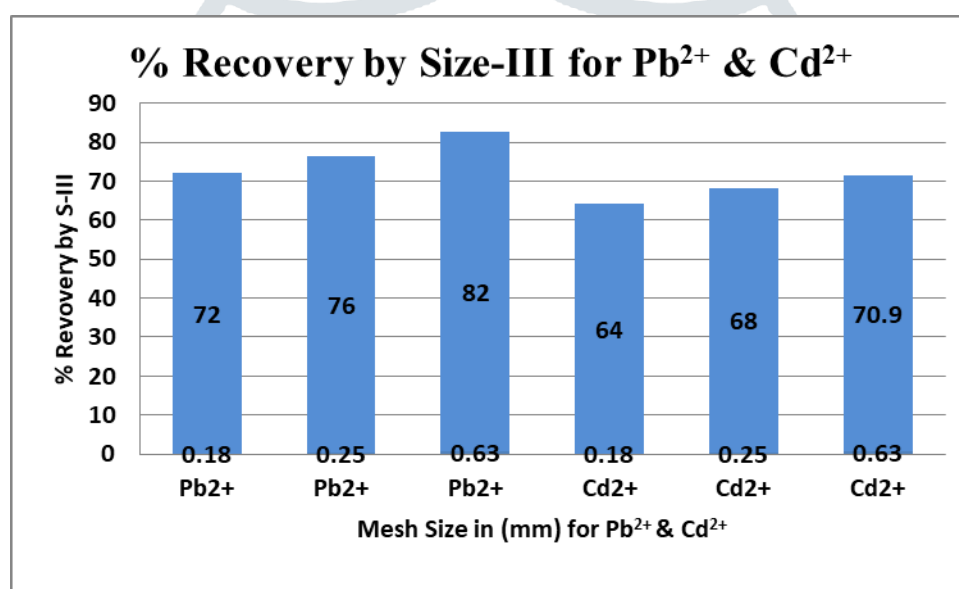


Fig-1: The % Recovery (Sorption) by Size-III for Pb<sup>2+</sup> and Cd<sup>2+</sup>

#### IV. CONCLUSION

- From total 25 mg of Pb<sup>2+</sup> and Cd<sup>2+</sup> taken in a mixture the quantity of metal ions recovered depends on 1. Nature of sorbent 2. Particle size.
- Pb<sup>2+</sup> and Cd<sup>2+</sup> can be separated quantitatively using Size-III (0.63 mm) adsorbent.
- Particle size affects separation and recovery but only to certain extent.
- 63 micron mesh particle when used for column packing there is maximum recovery of the metals Pb<sup>2+</sup> and Cd<sup>2+</sup>. (82 and 70.9 % respectively)
- Thus S-III (0.63 mm) is good stationary phase materials for quantitative Separation and recovery of heavy metals such as lead and cadmium from an admixture.

#### V. REFERENCES

1. Huang, C.P. and Wu, M.H. Chromium removal by carbon adsorption. Water. Res.1, 673, 1977.
2. Srinivasan, K. Balasubramaniam, N. Ind. J. Environ. Hlth. 30,376, 1988.
3. Singh, D. K. and Misra, N.K. IE (I). J. Chem. Eng., 70, 90, 1990.
4. Pandey, K.K., Prasad, G. J. Chem. Tech. Biotechnol, 34A, 367, 1984.
5. Sax, N.I.; Industrial pollution Van Nostrand reinhold company, New York, 1974.

6. Petrilli, F.L. and DeFlora, S. Toxicity and Mutagenicity of hexavalent chromium on *Salmonella typhimurium* Appl. Environ. Microbiol. 33805, 1977.
7. Huang, C.P. and Wu, M.H. Chromium removal by carbon adsorption. Water. Pollution. Control. Fed.47, 2437, 1975.
8. Srivastava, S.K., Tyagi, R. Water. Res., 23, 1161, 1989.
9. Balasubramaniam, M.R. and Murlishankar, I. Effluent, Ind. J. Technol. 25, 47, 1987.
10. Tchounwou, P. B., Yedjou, C. G., Patlolla, A. K. & Sutton, D. J. Heavy metal toxicity and the environment. Exp. Suppl. 101, 133–164, 2012.
11. Atieh, M. A., Ji, Y. & Kochkodan, V. Metals in the environment: Toxic metals removal. Bioinorg. Chem. Appl. 2017
12. Wu, X. et al. A review of toxicity and mechanisms of individual and mixtures of heavy metals in the environment. Environ. Sci. Pollut. Res. Int. 23, 8244–8259.
13. A. Mohammad, N. Fatima, TLC separations of some metal ions with aqueous and aqueous organic solvent systems containing formic acid, Chromatographia, 22 (1986) 109-116.
14. R.L. Smith, D.J. Pietrzyk, Liquid-Chromatographic Separation Of Metal-Ions On A Silica Column, Anal. Chem., 56 (1984) 610-614.
15. K.T. Denblyker, J.K. Arbogast, T.R. Sweet, High-Performance Liquid-Chromatography Of Metal-Ions On A Bonded Stationary Phase, Chromatographia, 17 (1983) 449-450.
16. K.T. Denblyker, T.R. Sweet, Thin-Layer Chromatography Of Metal-Ions On Bonded Stationary Phases, Chromatographia, 13 (1980) 114-118.
17. M.M. Hassanien, I.M. Kenawy, M.R. Mostafa, H. El-Dellay, Extraction of gallium, indium and thallium from aquatic media using amino silica gel modified by gallic acid, Microchimica Acta, 172 (2011) 137-145.
18. J.B. Henry, T.R. Sweet, Chemically-Bonded Stationary Phases For Thin-Layer Chromatography Of Metal-Ions, Chromatographia, 17 (1983) 79-82

