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Removal and recovery of heavy metals from mixture of Ni+2 and Pb+2 by Sulphonated and Nitrated Biomass of *Cicer arientinum* Column studies

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Abstract

The powdered biomass of Cicer arientinum has been converted in to cheap carbonaceous adsorbent by AR Sulphuric acid and AR. Nitric acid treatment. This adsorbent exhibits a good sorption potential for heavy metals like Ni^{2+} and Pb^{2+} from their mixture. Present work deals with removal and recovery of heavy metals like Ni^{2+} and Pb^{2+} from their mixture and chemical regeneration of the adsorbent material.

Key words: Column studies, Adsorption, adsorbent, Sulphonated material S-III, Nitrated material S-IV, Cicer arientinum.

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INTRODUCTION

Separation and recovery of these metals is also a matter of great concern for environment. This type of separation could be achieved by subjecting the effluents to column separation. For using an adsorbent as a stationary phase in the column the particles should have a specific size. The use of various adsorbents such as activated carbon[1], rice husk carbon[2], saw dust[3] and fly – wollastonite[4] have been documented for the removal of Cr (VI) from aqueous solutions. The toxicity of hexavalent chromium from the discharge of various industrial wastes is well reported[5-7] Srivastava have studied adsorption of heavy metal ions on carbonaceous material developed from waste slurry of fertilizer plants[8] . Balsubramanium have reported utilization of fly ash and waste tea leaves ash as decoloursing agents for dye effluents[9]. The presents study reports work on removal and recovery of heavy metals like Ni^{2+} and Pb^{2+} from their mixture. Our results on S-III and S-IV indicated that they were good adsorbents for Hg^{2+} and Pb^{2+},

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therefore trials were also taken to test their uptake capacity for nickel. These experiments gave promising results. As a further extension of this work we have tried to select particles of S-III and S-IV having appropriate size and carried out column chromatographic studies. Preliminary experiments on column chromatographic studies using S-III and S-IV as stationary phase materials have been carried out for the separation and recovery of Ni^{2+} and Pb^{2+} from their mixture. A large number of heavy metals have been recovered from effluents as well as from waste water by using synthetic organic, inorganic ion- exchangers, column methods. The details of these methods are described in many standard text books and in reviews. The distribution of metals on the ion exchanger can be modified by complex formation since the process of separation is not selective. However a number of separations could be achieved by complex formation due to enhanced selectivity. The most common example of selectivity is found with anionic chlorocomplexes of metal which are separated from hydrochloric acid solution by quaternary amine exchanger. Every metal has its own characteristic dependence of sorption on hydrochloric acid solution by quaternary amine exchanger. Every metal has its own characteristics dependence of sorption which makes an almost infinite variety of column chromatographic separations. Mishra have studied Fe (OH) $_3$ loaded marble as an adsorbent for the removal of phenolic compounds from aqueous solution. The adsorbed compounds have been quantitatively eluted with 1M NaOH.

MATERIALS AND METHODS

2.1 Materials and Reagents Agro-waste of *Cicer arientinum* AR. Sulphuric acid and AR. Nitric acid, HCl, Column Chromatographic, technique , complexmetric titration

2.2 Sample Preparation: Agro-waste of *Cicer arientinum* (Horse Bean) was dried powdered, subjected to simple chemical treatment with AR. Sulphuric acid and AR. Nitric acid and then washed repeatedly till free from acid and dried at 105^0C. The dried material was passed through sieves of 63 micron mesh ,25micron mesh,18 micron mesh size to get S-III and S-IV respectively of different particle size.

2.3 Column Preparation: Stock solutions of Ni^{2+} and Pb^{2+} of strength of 1mg/ml were prepared using the nitrates of the two metals. The column studies were carried out by using down flow method. For these experiments 20 gm of S-III of different particle size was suspended in distilled water, stirred for about 15 minutes and finally transferred into a glass column of length 30 cm and inner diameter 30 mm. A glass wool plug was kept at the bottom of the column to avoid the loss of adsorbent with the liquid flow. 50 ml of solution containing Ni^{2+} and Pb^{2+} was fed in to the column at the flow rate 5 ml/min.

2.4 Column Separation: The exhaustive capacity was determined by collecting 50 ml fraction of the eluent after break through. Similar experiments were carried out with different particle size of S-III and S-IV. The amount of metal ions retained by the sorbent was found out. The sorbed metal ion Ni^{2+} and Pb^{2+} loaded on the column were collected until the entire metal ion was completely removed using HCl and HNO_3 as eluents. The amount of metal ion extracted from the sorbent after elution was determined complexometrically. The amount of metal ion retained by sorbent and recovered after elution, from columns of S-III and S-IV of the different particle sizes (0.18, 0.25 and 0.63 micron) were calculated.

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RESULTS AND DISCUSSION

The results in **Table-1** indicate the effect of particle size on the recovery of the metals. As the particle size increases the % recovery also increases from 88 to 100%. For S-III and S-IV the recovery is 100% with (0.63mm) smallest sized particles. The % recovery **Table-2** of Ni^{2+} is in range between 84 % to 88 % indicating a slight increase with decrease in particle size of S-IV. Pb^{2+} shows a similar tendency on both exchangers S-III and S-IV and maximum recovery is 100 % and 96% respectively. A slight but distinct amount of Ni^{2+} and Pb^{2+} appears to be retained on the column containing S-IV. These results indicate that Ni^{2+} and Pb^{2+} can be separated and recovered quantitatively using S-III and S-IV as sorbents. The present study clearly indicates that S-III and S-IV are good materials for column separation of heavy metals viz Ni^{2+} and Pb^{2+} in quantitative yields.   Comparatively it was found that sorbent S-III have good removal and recovery than sorbent S-IV probably this may be due to strong oxidizing property of nitric acid. However further study is needed which would throw light upon the utility of these materials for recovery of heavy metals and also precious metals.

Fig-1   The % Recovery (Sorption) by S-III for Ni^{2+} and Pb^{2+}
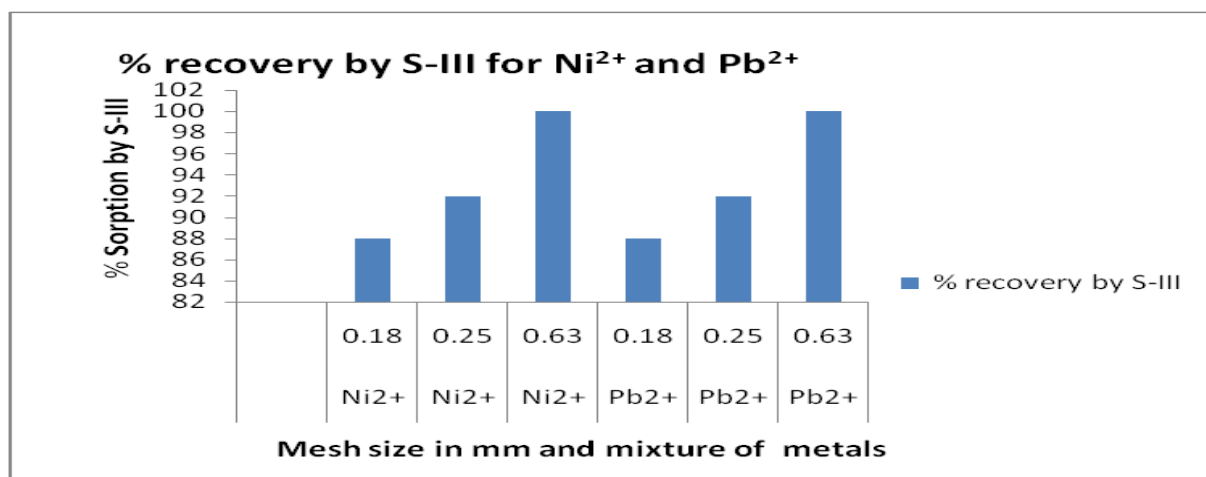
Fig-2 The % Recovery (Sorption) by S-IV for Ni^{2+} and Pb^{2+}

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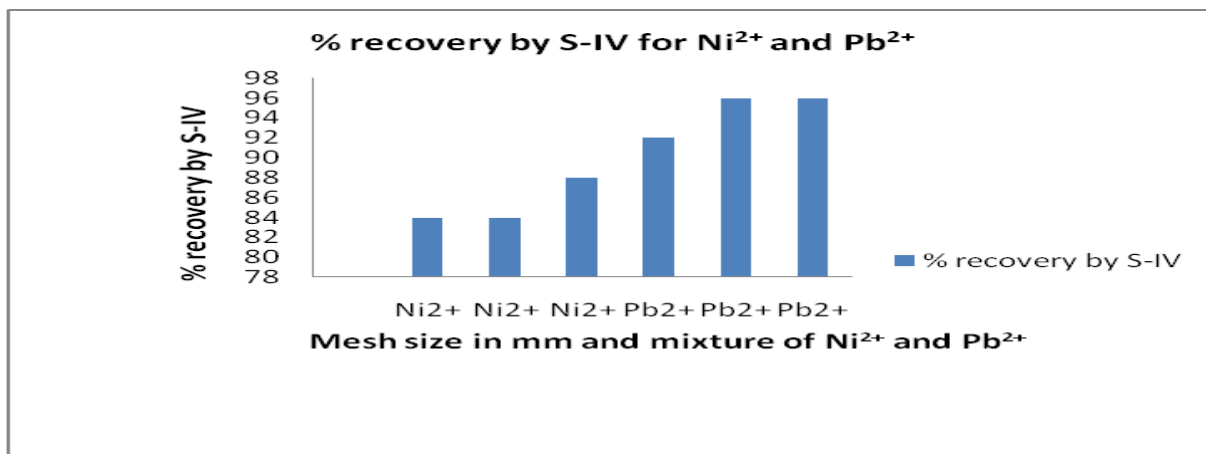
Fig-3 Comparison of % recovery (Sorption) by S-III and S-IV for Ni^{2+} and Pb^{2+}
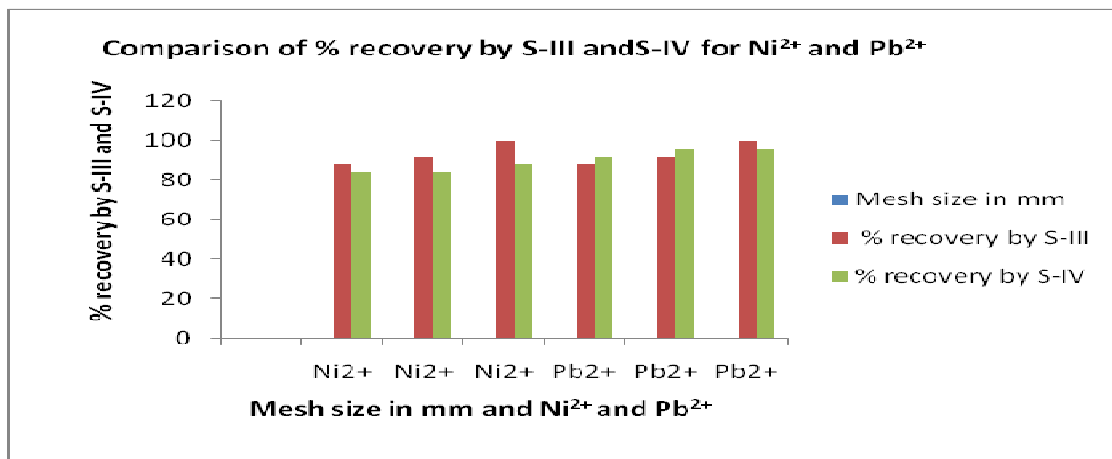
Table-1: The % Recovery (Sorption) by S-III for Ni^{2+} and Pb^{2+}

Metal ion	Amount sorbed (mg)	Amount recovered by S-III (mg)	% recovery by S-III	Mesh size in mm
Ni^{2+}	25	22	88	0.18
Ni^{2+}	25	23	92	0.25
Ni^{2+}	25	25	100	0.63
Pb^{2+}	25	22	88	0.18
Pb^{2+}	25	23	92	0.25
$Pb2^{+}$	25	25	100	0.63

Table-2: The % Recovery (Sorption) by S-IV for Ni^{2+} and Pb^{2+}

Metal ion	Amount sorbed (mg)	Amount recovered by-IV (mg)	% recovery by S-IV	Mesh size in mm
Ni^{2+}	25	21	84	0.18
Ni^{2+}	25	21	84	0.25
Ni^{2+}	25	22	88	0.63
Pb^{2+}	25	23	92	0.18
Pb^{2+}	25	24	96	0.25
$Pb2^{+}$	25	24	96	0.63

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CONCLUSION

- Out of 25 mg of Pb $^{2+}$ and Ni^{2+} taken in an admixture the amount of metal ions recovered depends on 1. Nature of sorbent 2. Particle size.
- Ni^{+2} and Pb^{+2} can be separated quantitatively using S-III and S-IV
- The surfaces of S-III and S-IV may not be very much different as there is only a slight difference in amounts of metal ions recovered**.**
- 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 Ni^{+2} and Pb$^{+2.}$ (96 and 100 % respectively)
- Pb $^{2+}$ and Ni^{2+} can be separated quantitatively ,Particles of S-III and S-IV Obtained by sieving through 63mm mesh when used for packing a column give 100% recovery on both metals Pb $^{2+}$ and Ni^{2+}
- Thus S-III and S-IV are good stationary phase materials for quantitative Separation and recovery of heavy metals such as nickel and lead from an admixture.

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