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ANALYTICAL APPLICATIONS OF SOME THREE COMPONENT CATION EXCHANGERS BASED ON TIN (IV) AND ANTIMONY(III)

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ABSTRACT

The distribution coefficient (K_d) of some metal ions in distilled water were calculated for three different cation exchangers namely tin(IV) tungstosilicate, antimony(III) iodostannate and tin(IV) vanadomolybdate. With the help of this study, binary separations of some important metal ion pairs were achieved. The effective separation of Ca^{2+} and Mg^{2+} , which causes hardness of water, was achieved. These cation exchangers were also employed in the removal of transition metal ions from their aqueous solutions. The column method of separation using these exchangers is simple, rapid and effective.

Keywords: *Distribution Coefficient, Cation Exchanger, Binary Separation, Hardness of Water, Transition Metal Ions*

INTRODUCTION

The analysis of trace elements in natural water, wastewater, biological, industrial and geological samples in complex mixtures is the challenging problem in analytical chemistry. Some of these elements are very harmful to flora and fauna. Therefore, it is necessary to remove such elements and it can be done by using some separation techniques. Thus separation is an important part of chemical analysis (Bhawna *et al.*, 2006).

Separation techniques can be considered to fall into two main categories. The one is bulk separation which involves large scale separations of one component from another. Other Category is the instrumental separations in which the amount of the analyte involved are too small to be physically observed (Mendham *et al.*, 2003). Some common separation techniques relating to the above stated categories are fractional distillation, crystallization, precipitation, solvent extraction, ion exchange, chromatography.

Ion exchange technology is most versatile technology in the field of separation science. At present it is a standard analytical tool and is widely used in inorganic, organic and biological separations. The ion exchange technique can be applied to both macro as well as micro-analysis and may serve even for the routine separation. The use of ion exchange material may provide pure water and may be useful for the recovery/ removal of metals.

Ion exchange may be defined as the reversible interchange of ions between a solid phase (ion exchange material) and a liquid phase in which there is no permanent change in structure of the exchanger (Jacob, 1969).

The early ion exchangers were silicates, fuller's earth and synthetic aluminium silicates e.g.-zeolites. Tree bark substrates were reported to remove efficiently, the heavy metals from waste water to below the product discharge limits. The exchange of calcium from soil to form calcium aluminosilicates indicated the phenomenon of simple ion exchange.

In the last forty years, there has been a great upsurge in the researches on synthetic inorganic ion exchangers. The emphasis has been given to the development of new materials possessing high chemical stability, high thermal stability, reproducibility in ion exchange behavior and selectively for certain metal ions are important from analytical and environmental point of view (Amphlett, 1958).

When a cation exchanger containing mobile cations (A^+) is brought into contact with a solution containing cations (B^+), the cations (B^+) diffuse into the exchanger structure and cations (A^+) diffuse out until equilibrium is attained. This is how the cation exchanger works. The solid and solution then contain both cations A^+ and B^+ in numbers that depends on the position of equilibrium. A cation exchanger can

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work by employing different operations. During our experimental work, we performed the column operation. In column operation, the column acts as a long series of solution exchanger equilibration stages. Thus it is an operation which involves repetitive equilibria (William, 1970).

Based on tin(IV) and antimony(III), some reported three component ion exchangers as shown by the literature are stannic silicomolybdate (Nabi and Amjad, 2006), stannic arsenosilicate (Varshney et al., 1984), stannic molybdophosphate (Marageh et al., 1999), stannic phosphotungstate (Susheel et al., 2007) and antimony tungstovanadate (Janardanan and Aravindakshan, 2001).

We synthesized three cation exchangers out of which two are based on tin(IV) (tin(IV) tungstosilicate and tin(IV) vanadomolybdate) and one is based on antimony(III) (antimony(III) iodostannate). These ion exchanges are three component inorganic cation exchangers. These exchangers have different affinity for different metal ions. Thus they can serve for the separation of different metal ions. The affinity of the exchanger for the metal ions is determined by calculating the distribution coefficient (K_d) values with the help of complexometric titration. The exchanger is most selective for the metal for which K_d value is maximum.

Ion exchange finds several applications in analytical chemistry. Ion exchange process is applied in several cases for separation of ions. Ions that interfere in many analytical procedures may be removed. A solution containing ions of the same nature of charge can also be separated by choosing an exchanger having exchange affinity for the ions to be separated. The analytical applications of ion exchange materials continue to increase (Verma, 2000). Some important applications of ion exchanger are –

- (1) Binary separation of metal ions.
- (2) Water softening
- (3) Removal of transition metal ions.

(1) Binary Separation of Metal Ions

This technique is used extensively for separation of metal ions from large volumes of natural and waste water. The total free metal ion content of a water sample is determined by passing the sample through cation exchanger and titrating the acid liberated with a standard alkali solution. For the separation of these metal ions, these ions are first concentrated on a suitable cation exchanger and then selectively eluted with the help of appropriate Eluent (De, 2006).

Table 1

Sl. No.	Metal	Major Sources	Harmful Effects on Man
1	Nickel	Diesel oil, coal, smoke, catalysts, steel and non, ferrous alloys.	Dermatitis, pneumonia.
2	Cobalt	Nuclear technology, manufacture of blue pigment for glass and china, textile mills.	Diarrhoea, low blood pressure, lung irritation, bone deformation paralyses.
3	Cadmium	Electroplating wastes, impurity in all products containing zinc.	Growth retardation, diarrhoea, bone deformation, kidney damage, anemia, injury of CNS.
4	Lead	Leaded gasoline, paints.	Anemia, vomiting, loss of appetite, convulsions, damage of liver, brain and kidney.
5	Copper	Metal planting, mining, mineral leaching.	Sphoradic Fever, Hypertension, Uremia, Coma.

Distribution studies revealed that the exchanger tin(IV) tungstosilicate is selective for Ni^{2+} and Co^{2+} . Antimony(III) iodostannate is selective for Cd^{2+} and Pb^{2+} whereas tin(IV) vanadomolybdate is selective for Ni^{2+} and Cu^{2+} .

Binary separation of those metal ions can be achieved for which the separation factor is greater than three.

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$$\text{Separation Factor } \alpha_B^A = \frac{K_d \text{ Value of metal ions A}}{K_d \text{ Value of metal ions B}}$$

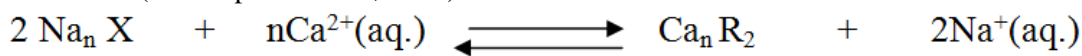
The separation factors are the guiding measures for the separation (Gupta *et al.*, 2000).

The sources and harmful effects of the above mentioned metals (Sharma, 2005) are given in table-1.

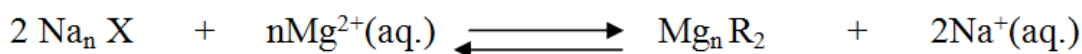
2. Water Softening

All water from any natural source contains certain amount of dissolved salts of calcium and magnesium. When these salts are contained in water in sufficient quantity so as to cause almost no frothing with the soap or detergent, it is called as the hard water and this is not suitable for household and industrial purposes (Shrivastava and Kamboj, 1999). The process of decreasing the hardness of water is called water softening. The softening proceeds, as is known by the filtration of water which contains calcium and magnesium ions through a bed of cation exchanger containing the material similar to the zeolites is based on the work by Gans which was published about 1905 (Robert).

When hard water is percolated through cation exchanger, Ca^{2+} and Mg^{2+} ions present in hard water may be removed (Gurdeep and Sham, 1995).



Ion exchanger	Hardness causing ion	Exhausted ion exchanger	Solution
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Ion exchanger	Hardness causing ion	Exhausted ion exchanger	Solution
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3. Removal of Transition Metal Ions

Transition metal ions (Co^{2+} , Ni^{2+} and Cu^{2+}) can be removed from their aqueous solutions with the help of cation exchangers. The determination of these metal ions in their aqueous solution was done qualitatively (Svehla, 1989) as well as quantitatively before and after passing through the column of cation exchanger. When an aqueous solution of a colored metal ion is passed through a bed of cation exchanger, the transition metal ion gets exchanged with the counter H^+ of the exchanger. As a result of this exchanged, the effluent becomes colorless containing only the counter H^+ whereas the exchanger acquires the color of that particular transition metal ion whose solution was passed through it and this can be visualized.

In the present work all the above stated processes were performed and observations were recorded. Following glassware's, reagents and chemicals were used to achieve the objectives.

Requirements

(A) Glasswares

Burette converted into column using glass wool, burette stand, chemical balance, oven, magnetic stirrer, graduated pipette, conical flask, beaker, funnel, measuring flasks, glass rod and test tubes with test-tube stand.

(B) Reagents and Chemicals

Lead nitrate, Bismuth nitrate, Sodium Hydroxide and EDTA were Qualigens product. All the acid i.e. Perchloric acid, Hydrochloric acid, Sulphuric acid and Nitric acid were also Qualigens product. Calcium carbonate, Ammonia and Hexamine were obtained from Sdfine chemicals. Chemicals such as Nickel ammoniumsulphate, Cadmium chloride, Magnesium acetate, Zinc acetate, Manganese acetate, Cobalt acetate, Copper acetate, Ammonium hydroxide, Ammonium nitrate and Ammonium chloride were also used in the experimental work. Different dyes used in the determination are Xylenol Orange, Fast Sulphon Black F, Bromopyrogallol Red and Eriochrome Black T.

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Experimental

(i) *Binary Separation of Metal Ions*

The value of separation factor for Ni-Cd, Ni-Mg, Ni-Pb and Ni-Mn metal ion pairs obtained for tin(IV) tungstosilicate were greater than three and their values are given in table-2 , therefore separations of Ni was done from Cd, Mg, Pb and Mn using tin(IV) tungstosilicate.

The separation of Ni-Cd was achieved on a 1 cm diameter glass column using the glasswool to support the exchanger bed. The column was washed thoroughly with distilled water. 500 mg of the tin(IV) tungstosilicate in H⁺ form was packed in the glass column. The solutions of nickel and cadmium ions each having the concentration of 0.01 M were prepared. 3% solutions of K_d values of metal ions were prepared to make the different combinations. Nickel and cadmium solutions were mixed together and diluted with distilled water. The diluted solution of nickel and cadmium ions was poured into the column. The solution was allowed to move through the column at the rate of 10 drops per minute and recycled three times. After this the column was washed with distilled water to rinse the sides of the column. The washing so obtained was passed through the same column. The adsorbed metal ions were then eluted with appropriate eluents one by one. The flow rate of the effluent was maintained at 1 ml per minute throughout the elution process. The nickel ions were eluted from the exchanger bed with the help of an eluent i.e. 0.001M HNO₃. After passing the eluent, the column was washed with distilled water and washing was collected in the same container containing the effluent. The concentration of Ni²⁺ in this effluent was determined by performing complexometric titration using Bromopyrogallol Red as an indicator.

The amount of Ni²⁺ obtained was compared with the Ni²⁺ amount taken initially to make the combination with Cd²⁺. The observations were recorded. Similarly cadmium ions were eluted with the help of 0.1M HNO₃+0.5M NH₄OH and then determined by the same type of titration using Eriochrome Black T as an indicator and the observations were recorded.

Similarly the separations of Ni-Mg, Ni-Pb and Ni-Mn metal ion pairs were achieved using the appropriate eluents with the help of tin(IV) tungstosilicate.

In the combination of Ni-Mg, 1.0M HNO₃ was used as an eluent for Ni²⁺ while 0.4M NH₄NO₃ was used as an eluent for Mg²⁺. The determination of Ni²⁺ and Mg²⁺ was done using Bromopyrogallol Red and Eriochrome Black T indicators respectively.

In Ni-Pb combination, Ni²⁺ and Pb²⁺ were separated using 0.1M HClO₄ as an eluent for Ni²⁺ and 0.1M HNO₃ as an eluent for Pb²⁺. Bromopyrogallol Red and Xylenol Orange were used as indicators for the determination Ni²⁺ and Pb²⁺ respectively.

Ni²⁺ and Mn²⁺ were separated from Ni-Mn combination using the different eluents. 1M NH₄Cl+0.1M HCl solution was used to elute the Ni²⁺ from the exchanger whereas 0.1M HCl was used to remove the Mn²⁺ from the ion exchanger. Both the effluents were collected in different containers.

Table 2: Separation Factor of Different Metal Ions for Tin(IV) tungstosilicate

Sample No.	Separation Factor	$\alpha_B^A = \frac{K_d \text{ Value of } A}{K_d \text{ Value of } B}$	Value of Separation Factor
1	α_{Cd}^{Ni}	$\frac{31.37}{8.49}$	3.70
2	α_{Mg}^{Ni}	$\frac{31.37}{5.11}$	6.14
3	α_{Pb}^{Ni}	$\frac{31.37}{7.05}$	4.45
4	α_{Mn}^{Ni}	$\frac{31.37}{5.83}$	5.40

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Table 3: Binary Separation Achieved With the Help of Tin(IV) tungstosilicate

Sample No.	Metal Ion Pair	Amount Loaded (µg)	Amount Found (µg)	% of Metal Ion eluted	% Error	Total Elution Volume	Eluent Used
1	Ni ²⁺	10095	9977	98.84	- 1.16	40 ml	0.001M HNO ₃
	Cd ²⁺	2023	1911	94.46	- 5.53	50 ml	0.1M HNO ₃ + 0.5M NH ₄ OH
2	Ni ²⁺	10095	10035	99.40	- 0.59	80 ml	1.0M HNO ₃
	Mg ²⁺	1507	1507	100	0.00	70 ml	0.4M NH ₄ NO ₃
3	Ni ²⁺	10095	10095	100	0.00	50 ml	0.1M HClO ₄
	Pb ²⁺	3108	2901	93.30	- 6.70	40 ml	0.1M HNO ₃
4	Ni ²⁺	10095	10153	100.57	+ 0.57	50 ml	1.0M NH ₄ Cl + 0.1M HCl
	Mn ²⁺	1922	1813	94.33	- 5.67	30 ml	0.1M HCl

The second ion exchanger (antimony(III) iodostannate) was also applied for binary separations. The separation factor of this exchanger for Pb-Ni, Cd-Mg, Cd-Ni, Co-Ni and Mg-Ni combinations was found to be more than three (table-4) therefore the metal ions in such combinations can be separated by employing the above stated ion exchanger.

Though elution process and the determination of metal ions was done in the similar way as given earlier, yet different indicators were used in different metal ion determinations.

Xylenol Orange indicator was used in complexometric titration to determine the amount of lead and cobalt. Two metal ions i.e. Cd²⁺ and Mg²⁺ were determined using Eriochrome Black T as an indicator. Bromopyrogallol Red was taken as indicator for the determination of nickel. The observations are recorded in table-5 from which the comparison of the loaded metal ion content and content of found metal ion can be done.

Table 4: Separation Factor of Different Metal Ions for Antimony(III) iodostannate

Sample No.	Separation Factor	$\alpha_B^A = \frac{K_d \text{ Value of A}}{K_d \text{ Value of B}}$	Value of Separation Factor
1	Pb ²⁺	<u>20.11</u>	9.62
	Ni ²⁺	2.09	
2	Cd ²⁺	<u>35.52</u>	4.47
	Mg ²⁺	7.94	
3	Cd ²⁺	<u>35.52</u>	17.00
	Ni ²⁺	2.09	
4	Co ²⁺	<u>12.75</u>	6.10
	Ni ²⁺	2.09	
5	Mg ²⁺	<u>7.94</u>	3.80
	Ni ²⁺	2.09	

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Table 5: Binary Separation Achieved With the Help of Antimony(III) iodostannate

Sample No.	Metal Ion Pair	Amount Loaded (µg)	Amount Found (µg)	% of Metal Ion Eluted	% Error	Total Elution Volume	Eluent Used
1	Pb ²⁺	7252	7252	100	0.00	40 ml	0.01M HNO ₃
	Ni ²⁺	1056	975	92.33	- 7.67	50 ml	0.1M HClO ₄
2	Cd ²⁺	5621	5845	103.10	+ 3.99	50 ml	0.1M HNO ₃
	Mg ²⁺	2187	2089	95.52	- 4.48	70 ml	0.4M NH ₄ NO ₃
3	Cd ²⁺	5621	5395	95.98	- 4.02	50 ml	0.1M HNO ₃ in 0.5M NH ₄ OH
	Ni ²⁺	1056	939.04	89.93	- 11.02	40 ml	0.001M HNO ₃
4	Co ²⁺	4419	4007	90.68	- 9.32	60 ml	0.5M HNO ₃
	Ni ²⁺	1056	982	93.00	- 7.00	40 ml	0.05M HNO ₃
5	Mg ²⁺	2187	2163	98.90	- 1.09	70 ml	0.4M NH ₄ NO ₃
	Ni ²⁺	1056	998	94.51	- 5.49	80 ml	1.0M HNO ₃

Similarly the third ion exchanger (tin(IV) vanadomolybdate) was also employed for binary separations of Ni-Pb, Zn-Co, Ni-Co, Ni-Mn, Ni- Mg and Cu-Co combinations as indicated by the value of separation factor for these metal ion pairs (table-6). The method was aforesaid. In the determination processes, Fast Sulphon Black indicator was used in the determination of copper while the other metal ions' determinations were done using the above stated indicators. Amounts of different metal ions found after elution were calculated. Percentage of metal ions eluted, their percentage error and their eluents are recorded in table-7.

Table 6: Separation Factor of Different Metal Ions for Tin(IV) vanadomolybdate

Sample No.	Separation Factor	$\alpha_B^A = \frac{K_d \text{ Value of A}}{K_d \text{ Value of B}}$	Value of Separation Factor
1	Ni ²⁺	<u>23.67</u>	4.65
	Pb ²⁺	<u>5.08</u>	
2	Zn ²⁺	<u>5.333</u>	26.40
	Co ²⁺	<u>0.202</u>	
3	Ni ²⁺	<u>23.67</u>	117.18
	Co ²⁺	<u>0.202</u>	
4	Ni ²⁺	<u>23.67</u>	58.44
	Mn ²⁺	<u>0.4048</u>	
5	Ni ²⁺	<u>23.67</u>	3.87
	Mg ²⁺	<u>6.109</u>	
6	Cu ²⁺	<u>12.25</u>	60.64
	Co ²⁺	<u>0.202</u>	

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Table 7: Binary Separation Achieved With the Help of Tin(IV) vanadomolybdate

Sample No.	Metal Ion Pair	Amount Loaded (μg)	Amount Found (μg)	% Metal Eluted	of Ion	% Error	Total Elution Volume	Eluent Used
1	Ni^{2+}	8217	8158	99.21		- 0.79	50 ml	0.1M HClO_4
	Pb^{2+}	2279	2279	100		0.00	40 ml	0.1M HNO_3
2	Zn^{2+}	1831	1766	96.45		- 3.55	40 ml	0.2M HClO_4
	Co^{2+}	707.16	650.23	91.94		- 8.05	60 ml	1.0M NH_4NO_3
3	Ni^{2+}	8217	8334	101.42		+1.42	40 ml	0.001M HNO_3
	Co^{2+}	707.16	707.16	100		0.00	60 ml	0.1M HNO_3 +0.5M NH_4OH
4	Ni^{2+}	8217	8275	100.71		+ 0.71	50 ml	1.0M NH_4Cl + 0.1M HCl
	Mn^{2+}	1540	1428	92.72		- 7.27	30 ml	0.1M HCl
5	Ni^{2+}	8217	8099	98.56		- 1.44	80 ml	1.0M HNO_3
	Mg^{2+}	1944	1871	96.24		- 3.76	70 ml	0.4M NH_4NO_3
6	Cu^{2+}	2923	2796	95.65		- 4.35	50 ml	0.2M HNO_3
	Co^{2+}	707.16	707	99.84		- 0.27	60 ml	0.2M HClO_4

(2) *Water Softening*

Hardness causing Ca^{2+} and Mg^{2+} were also removed with the help of all the three synthesized ion exchangers. Column method was used for the removal of ions. The hardness of the water samples was determined by complexometric titration method, in which Eriochrome Black T was used as an indicator. Similar methods were employed in all the three ion exchangers application for loading, eluting and determination of metal ions. Brief account of the procedure is given below-

Table 8: Removal of Ca^{2+} and Mg^{2+} with the help of Tin(IV) tungstosilicate

Sample No.	Metal Ion	Amount Loaded (μg)	Amount Found (μg)	% Metal Eluted	of Ion	% Error	Total Elution Volume	Eluent Used
1	Ca^{2+}	1603	1575	98.25		-1.74	50 ml	1.0M HNO_3
2	Mg^{2+}	1507	1507	100		0.00	50 ml	0.01 M HClO_4

Table 9: Removal of Ca^{2+} and Mg^{2+} with the help of Antimony(III) iodostannate

Sample No.	Metal ion	Amount loaded (μg)	Amount found (μg)	% metal eluted	of ion	% error	Total elution volume	Eluent used
1	Ca^{2+}	881.8	841.6	95.54		-4.46	50 ml	1.0M HNO_3
2	Mg^{2+}	2218	2188	98.92		-1.08	50 ml	0.01 M HClO_4

Table 10: Removal of Ca^{2+} and Mg^{2+} with the help of Tin(IV) vanadomolybdate

Sample No.	Metal ion	Amount loaded (μg)	Amount found (μg)	% metal eluted	of ion	% error	Total elution volume	Eluent used
1	Ca^{2+}	240.5	218	90.65		-9.35	50 ml	1.0M HNO_3
2	Mg^{2+}	1775	1750	98.59		-1.41	50 ml	0.01 M HClO_4

Definite volume of hard water sample was passed at a rate of 10 drops per minute through the column maintaining the bed of ion exchanger in the column. This process is repeated for three times. Hardness causing Ca^{2+} and Mg^{2+} loaded in the column were eluted using 1.0M HNO_3 and 0.01M HClO_4 as eluents

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respectively. The elution rate was maintained at 5 drops per minute. The eluted calcium and magnesium amount were determined as stated earlier. The results of the application of all the three ion exchangers are given in three tables (table-8, 9 and 10).

3. *Removal of Transition Metal Ions*

Applications of the same exchangers in removing the metal ions from different water samples was done following the same method i.e. column method. The determination of Co^{2+} , Ni^{2+} and Cu^{2+} was done to ascertain the amount of these ions in their aqueous solutions.

Table 11: Qualitative Tests for Transition Metal Ions for Tin(IV) tungstosilicate

Sample No.	Metal Ion	Color of the Salt Solution Before Passing Through Exchanger	Color of the Salt Solution After Passing Through Exchanger	Detection of Metal Ion in the Effluent
1	Co^{2+}	Pink	Colorless	Effluent + Sodium hydroxide solution → No precipitate(Co^{2+} absent)
2	Ni^{2+}	Green	Colorless	(a) Effluent + Sodium hydroxide solution → No precipitate(Ni^{2+} absent) (b) Effluent + Ammonia → No precipitate (Ni^{2+} absent)
3	Cu^{2+}	Blue	Colorless	(a) Effluent + Sodium hydroxide solution → No precipitate(Cu^{2+} absent) (b) Effluent + Ammonia → No precipitate (Cu^{2+} absent)

Table 12: Qualitative Tests for Transition Metal Ions for Antimony(III) iodostannate

Sample No.	Metal Ion	Color of the Salt Solution Before Passing Through Exchanger	Color of the Salt Solution After Passing Through Exchanger	Detection of Metal Ion in the Effluent
1	Co^{2+}	Pink	Colorless	Effluent + Sodium hydroxide solution → No precipitate(Co^{2+} absent)
2	Ni^{2+}	Green	Colorless	(a) Effluent + Sodium hydroxide solution → No precipitate(Ni^{2+} absent) (b) Effluent + Ammonia → No precipitate (Ni^{2+} absent)
3	Cu^{2+}	Blue	Colorless	(a) Effluent + Sodium hydroxide solution → No precipitate(Cu^{2+} absent) (b) Effluent + Ammonia → No precipitate (Cu^{2+} absent)

The method of determination was complexometric titration using Xylenol Orange, Bromopyrogallol Red and Fast Sulphon Black F for Co^{2+} , Ni^{2+} and Cu^{2+} respectively. The observations were recorded and the amounts are calculated. Different definite volumes of the three solutions were loaded on the three ion

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exchangers packed in three different columns. The flow rate of 10 drops per minute was maintained. The solutions were passed three times through the exchangers. The effluents of the three columns were collected in three different containers. The presence of the metal ions in all the containers was confirmed by performing qualitative analysis as given in table-11, 12 and 13. All the qualitative tests were found to be negative.

Table 13: Qualitative Tests for Transition Metal Ions for Tin(IV) vanadomolybdate

Sample No.	Metal Ion	Color of the Solution Before Passing Through Exchanger	Color of the Salt Before Passing Through Exchanger	Color of the Salt After Passing Through Exchanger	Detection of Metal Ion in the Effluent
1	Co ²⁺	Pink		Colorless	Effluent + Sodium hydroxide solution → No precipitate(Co ²⁺ absent)
2	Ni ²⁺	Green		Colorless	(a) Effluent + Sodium hydroxide solution → No precipitate(Ni ²⁺ absent) (b) Effluent + Ammonia → No precipitate (Ni ²⁺ absent)
3	Cu ²⁺	Blue		Colorless	(a) Effluent + Sodium hydroxide solution → No precipitate(Cu ²⁺ absent) (b) Effluent + Ammonia → No precipitate (Cu ²⁺ absent)

For quantitative determination of metal ions, suitable eluents were passed through all the columns containing loaded exchangers. The eluents used to elute the loaded metal ions along with their volume are given in table-14. After elution process the amount of metal ions was determined by the same method as given above (table-15, 16 and 17).

Table 14

Sl. No.	Metal Ion	Eluent	Volume of Eluent Used
1	Co ²⁺	1.0M NH ₄ NO ₃	60 ml
2	Ni ²⁺	0.1 M HClO ₄	50 ml
3	Cu ²⁺	0.2M HNO ₃	50 ml

Table 15: Removal of Transition Metal Ions With the Help of Tin(IV) tungstosilicate

Sl. No.	Metal Ion	Amount Loaded (µg)	Amount Found (µg)	% of Metal Ion Eluted	% Error
1	Co ²⁺	4826	4714	97.56	-2.44
2	Ni ²⁺	10095	9982	98.88	-1.11
3	Cu ²⁺	1855	1715	92.46	-7.54

Table 16: Removal of Transition Metal Ions With the Help of Antimony(III) iodostannate

Sl. No.	Metal Ion	Amount Loaded (µg)	Amount Found (µg)	% of Metal Ion Eluted	% Error
1	Co ²⁺	4419	4066	91.32	-8.68
2	Ni ²⁺	1056	939	88.92	-11.07
3	Cu ²⁺	3711	3558	95.88	-4.12

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Table 17: Removal of Transition Metal Ions With the Help of Tin(IV)vanadomolybdate

Sl. No.	Metal Ion	Amount Loaded (μg)	Amount Found (μg)	% of Metal Ion Eluted	% Error
1	Co^{2+}	707.16	665.45	94.10	-5.89
2	Ni^{2+}	8217	8092	98.48	-1.52
3	Cu^{2+}	2923	2798	95.72	-4.27

RESULTS AND DISCUSSION

Binary separation on the basis of K_d value and separation factor for some metal ion pairs have been computed and are given in table-3 for tin(IV) tungstosilicate, table-5 for antimony(III) iodostannate and table-7 for tin (IV) vanadomolybdate.

In binary separation of Ni-Cd, amounts found of nickel and cadmium shows that almost all the nickel metal ions are eluted while 94.46% of cadmium ions are eluted showing the percentage error of -5.58%.

In Ni-Mg separation, the difference between loaded amounts and amounts found show that magnesium is 100% eluted with 0% error and nickel is eluted to 99.40% with -0.59% error.

Similarly Ni-Pb and Ni-Mn pairs separation results are satisfactory as nickel is removed 100%. It means in Ni-Pb combination, Ni can be removed completely from any sample. Again in Ni-Mn combination, Ni is completely removed while manganese removal is 94.33%.

These results can be compared with the previously reported binary separation results. Ni-Mg separation achieved with the help of stannic(IV) antimonite (Aditya, 2000) shows that it removes only 96.10% of magnesium whereas tin(IV) tungstosilicate removes 100% magnesium from the said combination.

Second ion exchange (antimony(III) iodostannate), separation results are also found satisfactory as lead from Pb-Ni combination was completely removed. Amounts of different metal ions separated from different combination given in table-5 are according to our expectations. The percentage error ranges from -11.02 to +3.99. On comparison with stannic(IV) antimonite, it was found that our ion exchanger(antimony(III) iodostannate) removes more percentage of magnesium from Ni-Mg combination.

Similarly the results of the third ion exchanger(tin(IV) vanadomolybdate) were analyzed. Separation of different combinations was quite successful. The exchanger removed different metal ions to different extent such as 650.23 μg Co^{2+} was removed out of 707.16 μg Co^{2+} while 8334 μg Ni^{2+} was removed out of 8217 μg Ni^{2+} . The removal is seen from 91.94% to 101.42%. The exchanger was found to be better than tin(IV) tungstate (Alpana and Uma, 2003), zirconium(IV) selenomolybdate and stannic(IV) antimonite. Percentage of cobalt removed by tin(IV) vanadomolybdate is more than the above stated reported ion exchangers. The superiority of the exchanger can be proved with other examples too.

Ion exchangers can be used as water softeners. The three synthesized ion exchanger tin(IV) tungstosilicate, antimony(III) iodostannate and tin(IV) vanadomolybdate can remove Ca^{2+} and Mg^{2+} from hard water and thereby helpful in water softening. The results are given in table-8, 9 and 10.

The result obtained for tin (IV) tungstosilicate implies that Mg^{2+} can be removed from hard water upto 100% and removal of Ca^{2+} is 98.25%. Water softening results obtained for antimony(III) iodostannate revealed that the Ca^{2+} and Mg^{2+} can be removed to a great extent i.e. 95.54% and 98.92% respectively with the help of this exchanger. With the help of tin(IV) vanadomolybdate, the percentage of Ca^{2+} and Mg^{2+} eluted were 90.65% and 98.59% respectively.

The role of the ion exchangers is found to be useful in decontamination of the chemicals. Detection of the metal ions(qualitative analysis) made it possible to decide the determination process. Quantitative determination of metal ions in a sample helped in knowing the amount of metal ion present which in turn was helpful to decide the exchange process. The observation table clearly indicates that tin(IV) tungstosilicate and tin(IV) vanadomolybdate are nickel selective whereas antimony(III) iodostannate has more affinity for copper. Tin(IV) tungstosilicate, antimony(III) iodostannate and tin(IV) vanadomolybdate were found to be able to decontaminate 97.56%, 94.10% and 92.46% of cobalt respectively. This show the decreasing order of affinity of the exchangers towards cobalt.

Research Article

Conclusion

Synthetic ion exchange is now the established material in analytical chemistry useful in separation technology particularly because of their high selectivity for metal ion and ease of preparation in a desired composition and of selected characteristics. Our ion exchanger possesses selectivity for heavy metals such as Pb, Cd, Cu that are highly toxic to most of the organism. The exchangers are also employed for the softening of water. Therefore from all the results obtained we may conclude that use of these ion exchangers is easy, effective & economical in the field of analytical chemistry.

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