

Synthesis, Characterization and Analytical Application of Antimony (III) Iodovanadate

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Abstract: Antimony (iii)iodovanadate has been synthesized under varying ratio of antimony trichloride, sodium metavanadate and potassium iodate solutions. The synthesized material shows ion exchange capacity 2.67meq/g for Na^+ ions. The material has been characterized on the bases of IR, XRD and TGA. The chemical stability of the material has been determined in acid and base solutions of different concentration. Thermal stability of this exchanger has also been determined. Distribution studies exhibit maximum K_d value for Ni^{++} and minimum for Pb^{++} . The utility of the exchanger is revealed from the binary separations. The exchanger has been employed to achieve Binary separations Ni-Zn, Ni-Pb, Pb-Zn and Pb- Mg pairs.

Keywords: Inorganic cation exchanger, Ion exchange capacity, Chemical stability, Thermal stability, Distribution coefficient, Biary Separation.

I. Introduction

The importance of synthetic inorganic ion exchangers has increased attention due to their high selectivity and stability at elevated temperatures¹. In recent years several theories for ion exchange equilibria have developed and tried on inorganic ion exchange materials². Ion exchange is a widely used technique for selective separation of ions from different solutions. A highly selective ion exchange process is required for the treatment of waste effluent as, in most cases, the toxic ion to be removed, exists at very low concentration together with other ions that exist at much higher concentration³. Analytical application in the field of separation science have been reviewed by Inczedy⁴ and Walton⁵. Antimonates of zirconium⁶, titanium⁷, tin⁸, cerium⁹, thorium¹⁰, tantalum¹¹ and antimony(III) tungstosilicate¹² have been synthesized and used for various separation in analytical chemistry.

The present work deals with the synthesis and ion exchange properties of antimony(III) iodovanadate. The material has been characterized on the basis of thermal studies, chemical stability, TGA, XRD, IR and distribution studies. The exchanger is found to be selective for Ni^{++} and Pb^{++} as per distribution studies done in the research laboratory. Antimony(III) based ion exchanger received attention because of their excellent ion exchange behavior. The reproducible behavior and ion exchange properties prove their significances. Their utility has been demonstrated for the binary separations of various metal ions. Antimony(III) iodovanadate has been used for binary separations of the following pairs: Ni^{2+} - Cd^{2+} , Pb^{2+} - Zn^{2+} , Ni^{2+} - Pb^{2+} and Ni^{2+} - Zn^{2+} . The exchanger was also used for the removal of metal ions from aqueous media. Qualitative analysis of certain metal ions solutions before passing through the exchanger and after passing through the exchanger was done to ensure the presence and absence of metal ions in aqueous solutions respectively.

II. Experimental

Reagents & Chemicals - Antimony tri chloride, sodium meta vanadate and potassium iodate (All CDH of AR grade) were used. All other reagents used were also of analytical or chemically pure grade.

Instrumentation - Oven NSW (INDIA) (model - I 43), ROTARY SHAKER TANCO, Magnetic stirrer with hotplate, MUFFL FURNACE (Digital temp controller) SHIVAKI T-701 and Toshniwal research p^{H} meter model p^{H} 110, Samson electronic weighing balance (Model - 300 D) were also used in the Research laboratory, chemistry department, Meerut College Meerut. TGA, XRD and IR were obtained from IIT Roorkee where Perkin Elmer (Pyris Diamond) in alumina pan with a current of nitrogen, Thermo Nicolet for TGA, Philips Analytical X-ray B.V. diffractometer for XRD and Thermo Nicolet IR-spectrophotometer for IR were available.

Synthesis

Six samples of Antimony(III)iodovanadate were prepared by mixing 0.05 M Antimony trichloride, 0.1M Potassium iodate and 0.1M Sodium metavanadate solutions in different volume ratios with continuous stirring. The pH was adjusted to 1 of all the six mixtures. The formed precipitates were allowed to settle over

night and then filtered and washed with distilled water. The precipitates of all the six ratios were dried in an oven at 40+ 1^oC. The dried materials were broken down into granules by placing them in distilled water just after removing them from the oven and finally converted to the acid form by immersing them in molar nitric acid solution which was intermittently replaced, washed with distilled water till the materials become free of acid and again dried at room temperature to make ready for characterization. Six yellow coloured Antimony (III)iodovanadate samples were obtained. The results are summarized in Table 1.

Determination of Ion Exchange Capacity

Ion exchange capacity (IEC) of all the six sample were determined by column method (Table 1). On the basis of IEC sample No. AIV 6 was selected for detailed study and therefore was synthesized in bulk by the procedure as adopted earlier. p^H titration method was also followed for the determination of IEC of the exchanger synthesized in bulk.

Column Method: - The ion exchange capacity of the selected sample was also determined for different metal ions by column method. 0.05g of Antimony (III)iodovanadate in the H⁺ form was packed in a glass column having a glass wool support at the base. A 0.1M solution of different metal ions were passed through the bed of ion exchanger slowly by adjusting the effluent rate at 9-10 drops per minute. The collected effluents were titrated against a standard sodium hydroxides solution/ Ethylenediamine tetraacetic acid solution using appropriate indicators. The result is summarized in Table 2.

p^H Titration Method :- pH titration was also performed by batch method. Equal portions of the exchanger were equilibrated with different volume mixture of 0.1M NaCl and 0.1M NaOH solutions. The graph was plotted between pH and hydrozylion concentration (Fig1).

Thermal Studies

Ten equal portions of Antimony(III)iodovanadate were heated at different temperatures in muffle furnace for 1 hr and then the ion exchange capacity was determined of all the samples using a standard sodium hydroxide solution. The results are summarized in Table 3. The graph was plotted between temperature and I.E.C. (Fig 2). TGA of the exchanger shows the loss of water molecules and other components at different temperatures. Finally the weight of the material become constant (Fig. 3).

Chemical Stability

Equal amounts of the exchanger were kept in equal volumes of the various mineral acid solutions, base solutions and salt solutions of different concentrations for 24 hrs with intermittent shaking. The results are summarized in Table 4.

Distribution Studies

Distribution studies were carried out for several metal ions by batch process [9]. Seven equal portions of the exchanger in the H⁺ form were equilibrated with equal volumes of solutions of different metal salts in aqueous medium. The metal ion concentrations in the solution before and after equilibrium were determined by titrating against EDTA solution. The distribution coefficient values were calculated using the following formula.

$$K_d = \frac{I - F}{F} \times \frac{V}{W} \text{ (ml / g)}$$

Where

Kd	=	Distribution Coefficient
I	=	Initial amount of the ion in the solution phase.
F	=	Final amount of the ion in the Solution phase.
V	=	Volume of the solution (ml)
W	=	Weight of the ion exchanger (g).

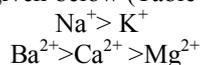
The results are summarized in table 5.

Applications Binary Separation Of Metal Ions

Important quantitative separations of metal ions were performed using column containing 200 mg of the material. The column was washed with demineralized water and the mixture to be separated was loaded on it in solution form. Recycling was done 3 times to ensure complete adsorption of the cations on the column bed. The metal ions were eluted at a flow rate of 3 drops min⁻¹ using appropriate eluents. The pairs were selected on the basis of Kd values obtained. The metal ions in the effluent were determined quantitatively by EDTA titration using suitable indicator (Table. 6)

III. Result And Discussion

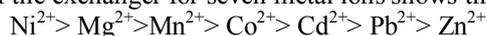
The properties of inorganic ion exchanger very widely changed when the conditions of their preparation are changed [10]. Six different samples of Antimony(III) iodovanadate have been prepared under varying ratios (Table 1). The synthesized Antimony(III) iodovanadate exhibits good ion exchange capacity (Table 2). Antimony(III) iodovanadate shows the maximum ion exchange capacity for Na⁺ ion is found to be 2.67 meq/g. The sequence of ion which exchange capacity for the alkali and alkaline earth metal ions as shown by the ion exchanger by column method is as given below (Table 2).



The study including the effect of temperature on ion exchange capacity of Antimony (III) iodovanadate shows that the ion exchange capacity decreases with increase in temperature.

The exchanger is found to be stable in water. Partly dissolved in 2MH₂SO₄, 2MNaOH, 2MNH₄Cl, 4M H₂SO₄, 4MHNO₃. Complete dissolve in 4M HCl solutions.

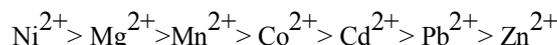
The distribution study of the exchanger for seven metal ions shows the affinity in the following order:



This shows that the exchanger is selective for nickel ions.

X-ray powder diffraction pattern shows the exchanger to be crystalline in nature, (fig.4). 4M HNO₃ and Completely dissolved in 4M HCl solutions.

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IR of the exchanger is given in fig. 2.6. Peaks are at 3433 cm⁻¹ for water molecules, 2450.65 cm⁻¹ for metaloxide, 1633.30 cm⁻¹ for H-O-H bonding, 756.96 cm⁻¹ for iodate, 450.22 for vanadium oxide. The presence of water molecules is well evident from the figure 5.

The present study shows that the exchanger is suitable for the binary separation of Ni²⁺-Zn²⁺, Ni²⁺-Cd²⁺, Pb²⁺-Zn²⁺ and Ni²⁺-Pb²⁺ metal ion pairs. The result are shown. (Table 6.)

IV. Conclusion

The present study clearly indicates that the synthesized exchanger can be used successfully in acid solutions of 0.05 Molar strength. The study also shows the selectivity of the exchanger towards nickel ions.

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TABLE-1SYNTHESIS OF ANTIMONY(III) IODOVANADATE

S. No.	Molar Conce			Mixing Ratio			pH of the mixture	Appearance of the precipitate	Appearance of beads after drying at 40+1°C	I.E.C. for Na ⁺ (meq gm ⁻¹)
	A	I	v	A	I	V				
1	0.05M	0.1M	0.1M	1	1	1	1	Lemon yellow	Lemon yellow	*
2	0.05M	0.1M	0.1M	1	2	2	1	Lemon yellow	Lemon yellow	*
3	0.05M	0.1M	0.1M	2	1	1	1	Lemon yellow	Lemon yellow	1.2
4	0.05M	0.1M	0.1M	2	2	1	1	Lemon yellow	Lemon yellow	1.6
5	0.05M	0.1M	0.1M	3	1	1	1	Lemon yellow	Lemon yellow	1.28
6	0.05M	0.1M	0.1M	3	2	2	1	Lemon yellow	Lemon yellow	2.67

A – Antimony trichloride Solution

I– Potassium iodate

V – Sodium metavanadate Solution

TABLE – 2 I.E.C OF ANTIMONY(III) IODOVANADATE FOR DIFFERENT METAL IONS

SL.No.	Cation	I.E.C. (meq gm ⁻¹)
1	Na ⁺	2.28
2	K ⁺	1.7
3	Mg ⁺⁺	1.16
4	Ca ⁺⁺	2.4
5	Ba ⁺⁺	2.6

Alkali earth metal ions – Na⁺ > K⁺

Alkaline earth metal ions – Ba⁺⁺ > Ca⁺⁺ > Mg⁺⁺

TABLE – 3 THERMAL STABILITY OF ANTIMONY(III) IODOVANADATE

SL.No.	Temperature (°C)	wt. before heating (g)	wt. after heating (g)	Loss in wt. (g)	Colour after heating	I.E.C. (meq gm ⁻¹) after heating
1	50 ⁰ C	0.50	0.50	No Effect	Yellow	2.67
2	100 ⁰ C	0.50	0.49	0.01	Yellow	2.55
3	150 ⁰ C	0.50	0.48	0.02	Yellow	2.50
4	200 ⁰ C	0.50	0.47	0.03	Yellow	2.40
5	250 ⁰ C	0.50	0.46	0.04	Yellow	2.35
6	300 ⁰ C	0.50	0.45	0.05	Yellow	1.89
7	350 ⁰ C	0.50	0.44	0.06	Yellow	1.82
8	400 ⁰ C	0.50	0.43	0.07	Yellow	1.77
9	450 ⁰ C	0.50	0.42	0.08	Brown	1.17
10	500 ⁰ C	0.50	0.41	0.09	Brown	0.80

TABLE – 4 CHEMICAL STABILITY OF ANTIMONY(III) IODOVANADATE

SL.No.	Solution	Effect of Solution on BIT				Colour	W ₁ = weight of IE before treating with solution W ₂ = weight of IE after treating with solution PD = Partly dissolved IE=Ion Exchanger ATS=Antimony(III)iodovanadate CD = Complete Dissolve
		W ₁	W ₂	Inference			
1	2 M H ₂ SO ₄	0.25	0.24	PD	Yellow		
2	4 M H ₂ SO ₄	0.25	0.23	PD	Yellow		
3	4 M HNO ₃	0.25	0.20	PD	Golden Yellow		
4	4M HCl	0.25	CD	CD	Yellow		
5	2 M NaOH	0.25	0.10	PD	Yellow		
6	2 M NH ₄ Cl	0.25	0.23	PD	Yellow		

TABLE – 5 DISTRIBUTION STUDIES OF ANTIMONY(III) IODOVANADATE

SL.No.	Cation	Taken as	Kd (ml/g)
1	Mg ²⁺	Chloride	90.52
2	Zn ²⁺	Sulphate	4.13
3	Cd ²⁺	Nitrate	18.4
4	Pb ²⁺	Nitrate	16.44
5	Ni ²⁺	Sulphate	111.25
6	Co ²⁺	Sulphate	33.12
7	Mn ²⁺	Sulphate	85.55

TABLE-6BINARY SEPERATION

Sl. No.	Sample No.	Seperation	Amount Loaded(μ g)	Amount Founed (μ g)	% of Metal Ion Eluted	% Error	Total elution volum (ml)	Eluent used
1.	AIV-6	NI Zn	1545 2731	1441 2875	93.26 105.27	+6.74 -5.27	50 30	1M NH ₄ cl+0.1M HCl 0.5 M NH ₄ Cl
2.	AIV-6	NI Cd	1545 7238	1572 7392	101.74 102.12	-1.74 -2.12	50 50	1M NH ₄ cl+0.1M HCl 0.1 M HNO ₃ +0.5 MNH ₄ OH
3.	AIV-6	Pb Zn	2731 990	2731 825	83.33 100.00	16.67 0.00	40 30	1M NH ₄ cl+0.1M HCl 0.5 M NH ₄ Cl
4.	AIV-6	Ni Pb	1545 990	1441 1155	93.26 116.66	+6.74 -16.66	50 40	1M NH ₄ cl+0.1M HCl 1M NH ₄ cl+0.1M HCl

Fig-1 pH titration curve of Antimony(III) iodovanadate

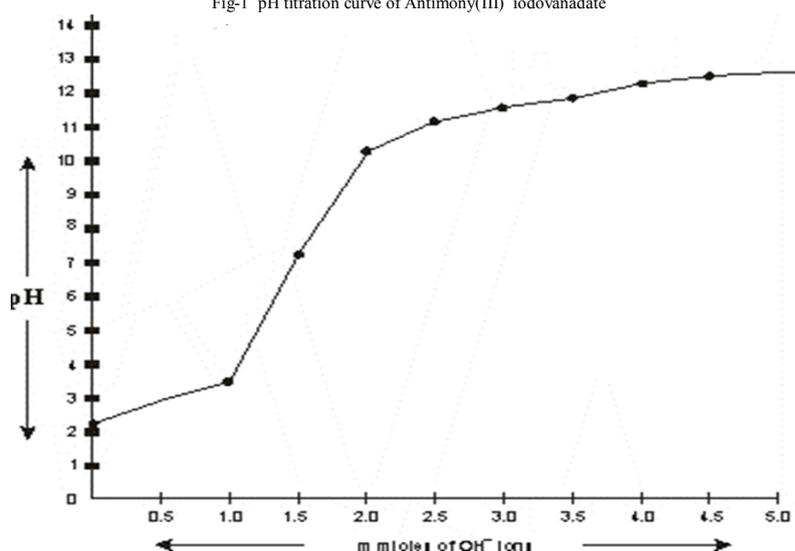


Fig-2

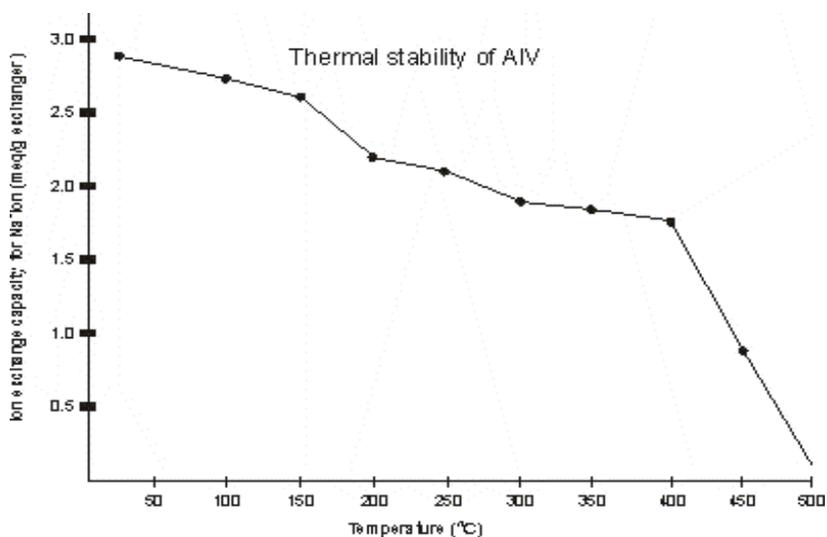


Fig: 3 TGA CURVE OF AIV

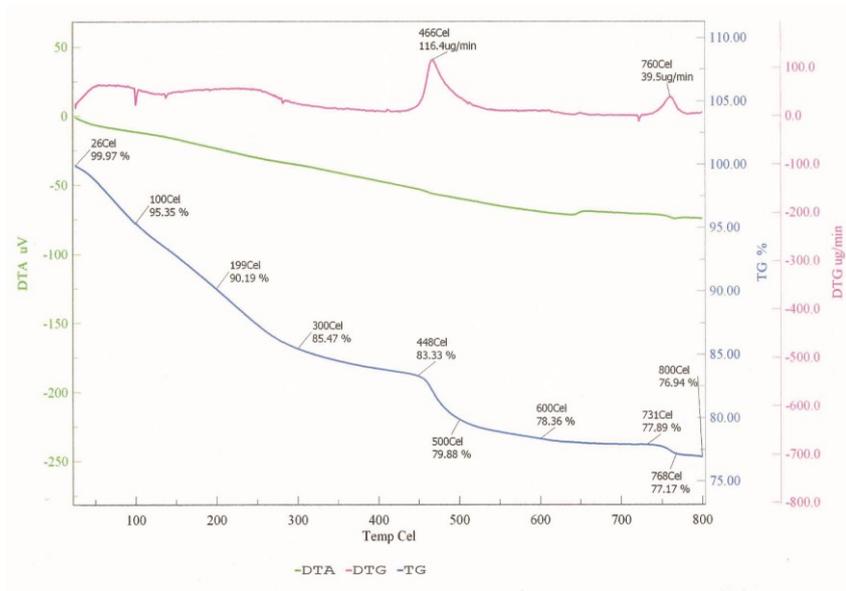


Fig: 4 IR SPECTRUM OF AIV

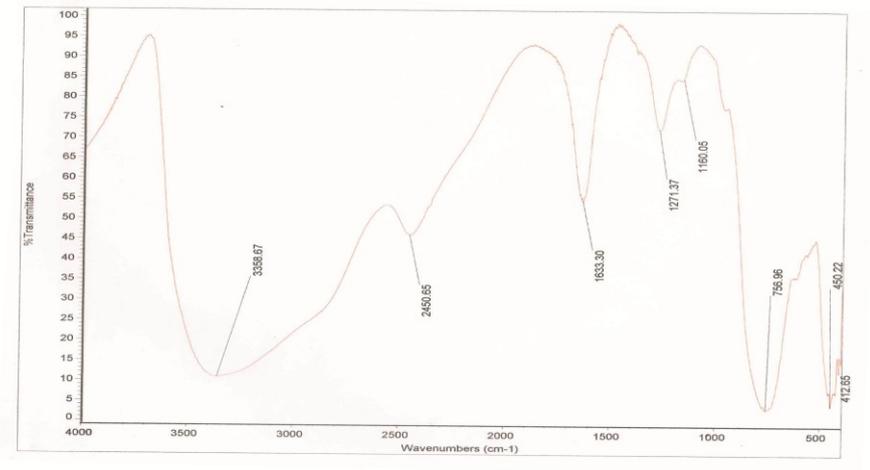
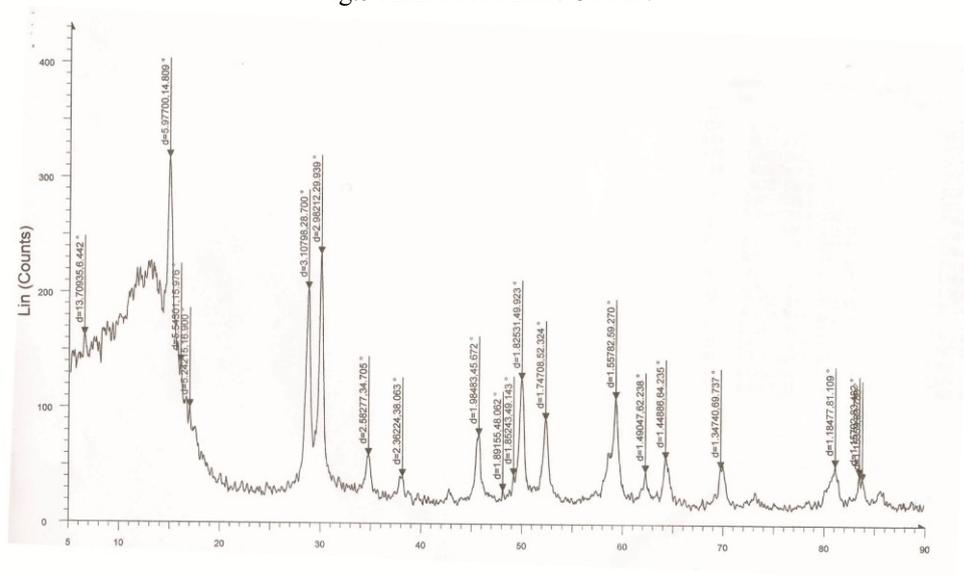


Fig:5 XRD PATTERN OF AIV



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