Synthesis and Ion Exchange Properties of Cerric Antimonate

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Introduction

During the last few decades new inorganic ion exchangers have been synthesised and studied. Despite the numbers of cerium salts (cerium phosphate and cerium arsenate . . .etc.) that have been prepared previously[1], no information concerning the synthesis of cerric antimonate was found in the literature.

However, antimonic acid was reported as inorganic ion exchange material[2]. Therefore, it was decided to synthesise cerric antimonate and study its analytical application. Such a study is considered important because of the limited knowledge of this exchanger. This paper is a preliminary study and summarises the results obtained on the selectivity of cerric antimonate for different number of metal ions other than alkali metal ions. Some important pairs of ions have been seperated on columns of cerric antimonate.

Experimental

Reagents: Cerric ammonium nitrate (BDH, AR) antimony trichloride (BDH AR) and ammonium solution (BDH AR) were used, and all the salts of metal ions used for distribution studies were of AR grade.

Apparatus : pH and distribution studies were made by using pH DIGI 520 meter and shaker, respectively. For seperations a column of glass of i.d. -0.8 mm with glasswool support was used.

Preparations : The samples of cerric antimonate were synthesised at different pH values as follows: 0.1M cerric ammonium nitrate was mixed with equal volume of 0.1 M $SbCl_3$ which was previously oxidized by adding conc. HNO_3. The mixing was done in 4M HCl to prevent any hydrolysis of $SbCl_5$. Ammonia solution was then added to the mixture with constant shaking to get the gel at the desired pH value.

Results : Some of the properties of cerric antimonate under study are summarised in Table 1.

Table 1

Distribution coefficient (Kd) of metal ions at different pH values on cerric antimonate.

Cation	pH 0	 3	 6–7
Mg^{++}	0.00	0.00	4.24
Ca^{++}	438.8	970	4750.00
Sr^{++}	111.4	27.6	146.70
Ba^{+++}	22	13	1050.00
Pb^{++}	7.5	138.9	0.00
Zn^{++}	0.00	3.5	32.50
Cd^{++}	0.00	24.1	89.50
Hg^{++}	116.7	290	1118.70
Al^{+++}	0.00	78.6	100.80
Fe^{+++}	0.0	75.5	111.80
Ga^{+++}	30.8	109.3	278.30
Y^{+++}	0.0	17.4	78.40
Ni^{++}	33.3	220	700.00
Co^{++}	0.00	2	45.80
Cu^{++}	7.5	330	1433.80
Bi^{+++}	33.3	900	0.00

Solubility : The samples synthesised at lower pH were found to be unstable in dilute acids and even water. However the samples prepared at high pH values were found to be highly stable in water and dilute acids up to 1M. The sample selected for distributions studies was very slightly soluble in more than 1M solutions of acids like HCl, H_2SO_4 and HNO_3.

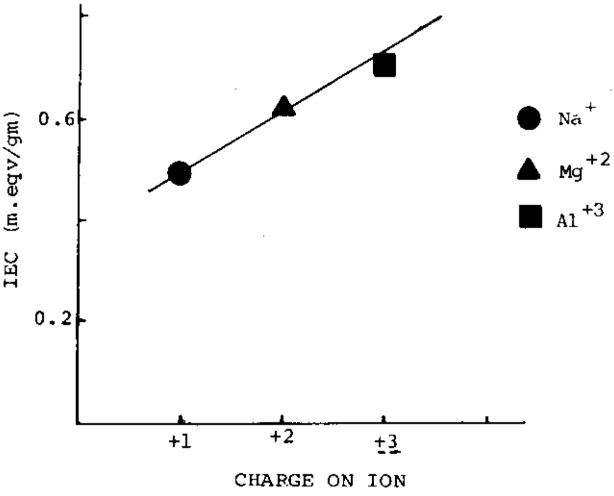


Fig. 1. IEC Vs. Charge on Ion

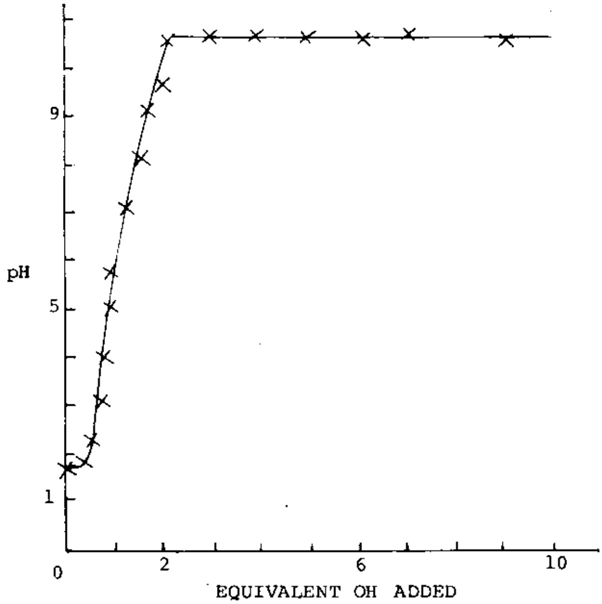
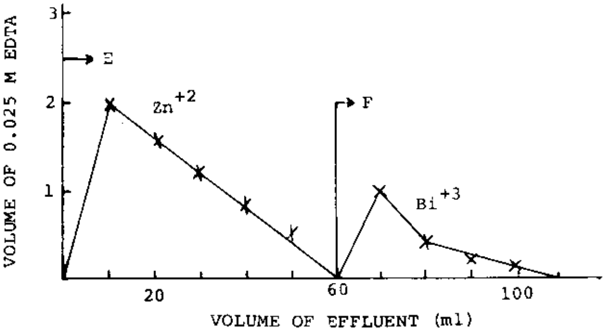
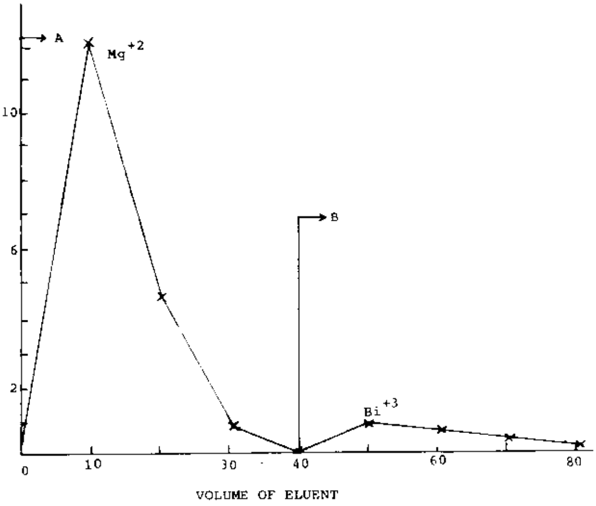


Fig. 2 pH titration Curve



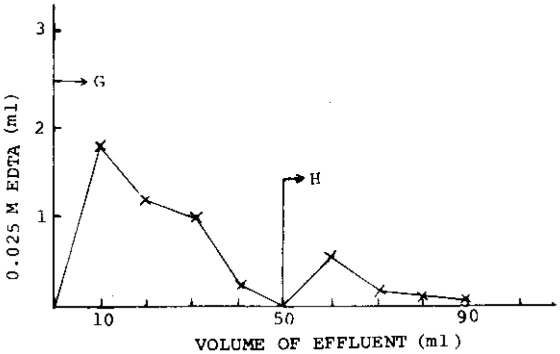
E=DISTILLED WATER
F=10% HNO_3

Fig. 3 Sepration of $Zn^{2+}-Bi^{3+}$ admixture



A = 0.1% NH_4Cl
B = 10% HNO_3

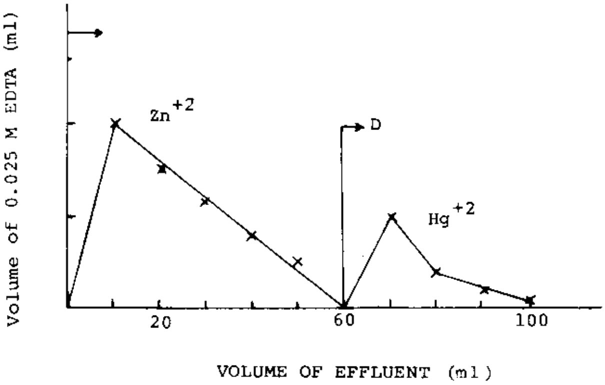
Fig. 4 Separation of $Mg^{2+}-Bi^{3+}$ admixture



G=0.05 M HNO_3

H=0.1 M NH_4NO_3+0.1 M HNO_3

Fig. 5 Separation of $Cd-Cu^{+2}$ admixture



C = H_2O
D = 2% HNO_3 + 5% NH_4NO_3

Fig. 6 Separation of $Zn^{+2}-Hg^{+2}$ admixture

Ion-exchange properties :

The cation exchange capacities for Na^+, Mg^{2+}, Al^{3+}, (Fig.1) pH titrations (Fig.2) and distribution studies were prepared by the method as reported earlier[3] by using the formula:

$$Kd = \frac{I-F}{F} \times \frac{50}{0.5} \ ml/g$$

Where I is the volume of 0.025M EDTA before equilibrium. F=Volume of EDTA (0.025M) after equilibrium for seperations: lg of exchanger in H^+ form was loaded in the column with glass wool support.

The quantitative estimates of all the cations was done by compleximetric titrations using Metall-achromic indicators[4].

The Ion exchange capacity shows a gradual increase with increase in the charge of the cations (Fig.1), which shows greater the charge and smaller the size of the ion more easily it can displace more H^+ ions in the exchanger.

pH titration curve (Fig.2) indicate that exchanger is monobasic acid. The distribution coefficient shows a regular trend at different pH values.

They are summarised in Table 1. The seperation factors have been calculated (table 2) and on the basis of seperation factors some good binary seperations have been achieved quantitatively on the column of the exchanger (Figs 3-6).

Discussion

Cerric antimonate shows some useful features as an cation exchanger. It has a high up take of many cations. The K_d values are higher than other reported exchanger[3] such as tin(IV) antimonate. The ion-exchange capacity as mentioned increases with increase of charge, such property was not being observed with other exchangers[3].

It can be deduced from previous studies[3] done on other antimonates that the structure of cerric antimonate is probably similar to that of tin(IV) antimonate and can be postulated as follows:

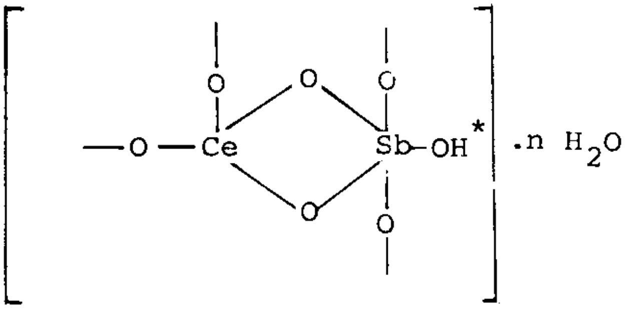


Table 2

Separation factors of some important pairs of ions on cerric antimonate

Pair	Separation factor
Bi^{+++},Zn^{++}	very large
Bi^{+++},Mg^{++}	very large
Ba^{++},Sr^{++}	very large
Mg^{++},Sr^{++}	very large
Hg^{++},Zn^{++}	34.4
Pb^{++},Co^{++}	very large
Bi^{+++},Al^{+++}	very large
Mg^{++},Ba^{++}	250
Ca^{++},Mg^{++}	1120.2
Ni^{++},Co^{++}	15.3
Cu^{++},Al^{+++}	13.3
Cd^{++},Cu^{++}	14.9

The hydrogen indicated by asterisk is considered responsibile for the cation-exchanger capacity. The importance of this study is reflected by the successful separation that was achieved using this synthetic exchanger.

In conclusion, this is a preliminary study and further studies using X-ray, thermal analysis and other techniques will be reported later.

Reference

1. V. Veselyu and V. Pekarek, *Talanta*, **19**, 262 (1972).
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3. M. Qureshi, V. Kumar Virendra and N. Zehra, *J. Chromatography*, **67**, 351–361 (1972).
4. R. Belcher, Nutten and A. MacDonald (Quantitative Inorganic Analysis), Butterworth, London (1975).