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Synthesis of Zirconium Phosphosilicate and Its Application as Inorganic Ion-Exchanger for Adsorption and Radiochemical Separation of Indium (III)

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Abstract: In the present investigation zirconium phosphosilicate has been synthesized and characterized for its composition. A rapid and selective method has been developed for the radiochemical separation of In (III) from other elements by adsorbing it on zirconium phosphosilicate as an ion-exchanger by a batch process. ^{114m}In is used as a tracer. The optimum time of contact and pH for the adsorption of 1 mg of In (III) has been found to be 5.0 min and 4.0 respectively. The adsorption was found to be maximum ($94.48 \pm 2.8\%$) with 200 mg of the exchanger. The interference of various anions in the adsorption of In (III) at optimum condition of adsorption has been studied. Adsorption of various other cation in presence of In (III) also has been studied. The distribution co-efficient values and the Decontamination factor has been evaluated.

Keywords: Batch Adsorption, Radiochemical Separation, Tracer Technique, Zirconium Phosphosilicate (ZPS), Distribution Co-Efficient.

I. INTRODUCTION

Various methods are available for the separation of trace elements present in diverse matrices. Of these, the use of inorganic ion exchanger is one of the most selective and reliable techniques [1]. Many inorganic ion exchangers have been synthesized and their use in various radiochemical separations explored [2], [3]. In radiochemical separation, inorganic ion exchangers are preferred due to their outstanding resistance to high radiation doses and temperature. In (III) is widely distributed in nature in trace concentrations. Commercially it is used in the production of gold based dental alloy; nuclear control rod alloys (Ag-In- Cd); solar cells and low-pressure sodium lamps etc. Indium phosphors are used in a computer monitor. The present work deals with the development of a rapid and selective radiochemical separation procedure employing zirconium phosphosilicate as an ion- exchanger for the separation of In (III) from other elements. ^{114m}In in was used as a tracer.

II. INSTRUMENTS AND REAGENTS:

All the chemicals used were of A.R. grade. ^{114m}In and the other radiotracers used for studying the adsorption of different elements were supplied by the Board of Radiation and Isotope Technology, Bhabha Atomic Research Centre, Mumbai. A solution of In (III) (10 mg/cm^3) was prepared by dissolving an appropriate amount of Indium chloride in distilled water.

Metal ion solutions of Co (II), Hg (II), Re (VII), Zn(II),Ca(II), Cd(II), Ir(IV), Na(I), K(I), Os(VI), Cr(III), In(III), Sc(III), Au(III), Pt(IV), Sn(II), Zr(IV), Ba(II), Cs(I), Rb(I) etc. (10 mg/ml concentration) were prepared by dissolving their appropriate salt in distilled water containing acid wherever required. The strength of the solutions was determined by the usual methods [4], [5]. Radioactivity of the beta emitters was measured by using GM counter whereas, the radioactivity of gamma emitters was measured by using gamma ray spectrometer having NaI (TI) detector coupled to the multichannel analyzer and the printer.

III. COMPOSITION OF ZIRCONIUM PHOSPHOSILICATE

The exchange was prepared by the method as reported by Naumann [6]. A known amount of zirconium phosphor silicate was dissolved in 1:1 HF solution which was warmed and diluted to 100 cm^3 . The weight percentage of ZrO_2 , P_2O_5 , and SiO_2 were determined gravimetrically (Table-I) and was found to be in close agreement with the values reported in the literature.

TABLE-I
Composition of Zirconium Phosphosilicate Ion-Exchanger

Composition	Weight %		
	ZrO ₂	SiO ₂	P ₂ O ₅
Observed	18.33	49.66	21.46
Expected	19.13*	48.18*	20.76*

* Result obtained by Baetsle et al [7].

IV. ADSORPTION OF INDIUM (III) (BATCH METHOD):

A 10 cm³ solution containing ^{114m}In and 1.0 mg In (III) carrier (pH adjusted to 4.0) was added to 200 mg of zirconium phosphosilicate exchanger for a contact time of 5.0 minutes. The activity in the effluent and on the exchange was measured on a multi-channel gamma-ray spectrometer, corresponding to the channel number of photopeak of ^{114m}In. The reproducibility of the adsorption was tested by evaluating the value five times.

V. RESULTS AND DISCUSSION

A. Effect of the Amount of Zirconium Phosphosilicate Exchanger

The adsorption studies were carried out at a pH of 4.0, as mentioned above, and the amount of the exchanger was varied between 100 and 500 mg. From Fig. I, it has been observed that the adsorption of 1.0 mg of In (III) was maximum (94.48%) with 200 mg of the exchanger. Hence, 200 mg of zirconium phosphor silicate was used in all further adsorption studies.

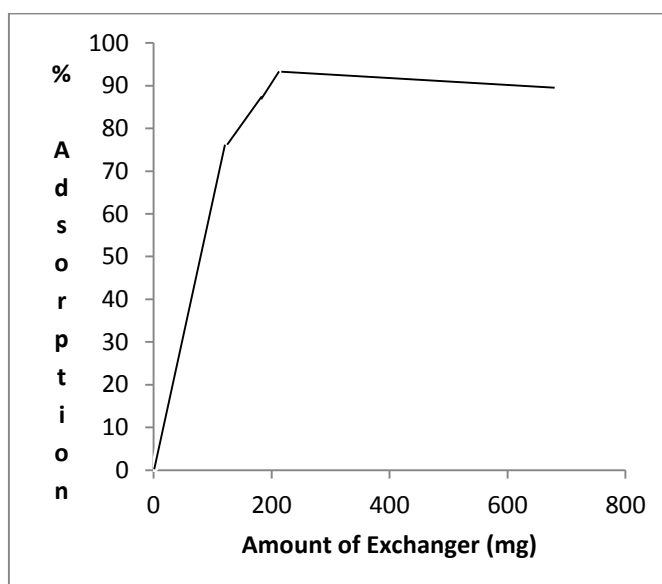


Fig. 1 Effect of Amount of Exchanger

B. Effect of Metal-Ion Concentration

The effect of concentration of In (III) on the adsorption was also studied by varying the concentration of In (III) between 0.5 mg and 5.0 mg. It has been observed that the adsorption of In (III) on 200 mg of zirconium phosphor silicate is maximum for 1.0 mg In (III) and remains practically constant thereafter up to 5.0 mg at pH 4.0.

C. Effect of pH

The effect of pH on the adsorption of In (III) revealed that the adsorption increases up to a pH of 4.0 and then remained practically constant. The results are shown in the Table-II. It has been observed that when the contact time was varied between 1.0 and 7.0 minutes, maximum adsorption was observed at 5.0 minutes, after which it practically remained constant.

Table-II Effect of pH

Amount of zirconium phosphosilicate	: 200 mg
Indium (III) concentration	: 1.0 mg + ^{114m} In
Time of contact	: 5.0 minutes
Temperature	: 28 ± 2°C

pH	% Adsorption
2.0	30.73
3.0	68.58
3.5	80.25
4.0	91.72
4.0	97.03
4.0	94.69
4.5	93.03
5.0	94.98
6.0	94.01

Reproducibility = $94.48 \pm 2.8\%$

D. Effect of time of contact

Effect of time of contact in adsorption of 1.0 mg of In (III) on 200 mg of zirconium phosphosilicate at pH 4.0 was also studied. From Fig. II, it has been observed that when the contact time was varied between 1.0 and 7.0 minutes, maximum adsorption was observed at 5.0 minutes, after which it practically remained constant.

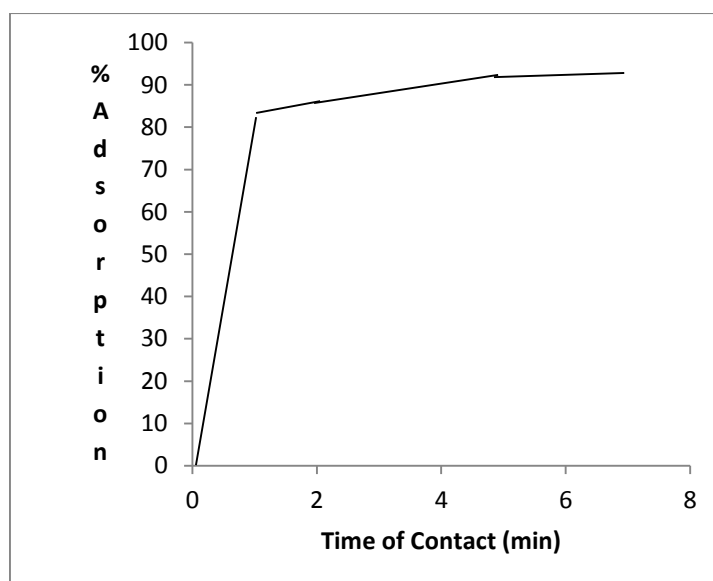


Fig. 2 Effect of Time of Contact

E. Interference Of Various Anions

The effect of different anions on the percentage adsorption of In (III) was investigated by taking anions as salts of Na^+ , K^+ , NH_4^+ . The results revealed that 100 mg each of bromate, EDTA, acetate, tungstate, carbonate, tartrate, borate, chlorate, oxalate and thiosulphate; 50 mg each of cyanide, sulphate, nitrate, thiocyanate, sulphate, metabisulphite, nitrate, phosphate and citrate; 25.0 mg of ferrocyanide and 10.0 mg each of molybdate, fluoride, iodide and bromide did not interfere in the adsorption of In(III). Chromate and dichromate gave the precipitation under the experimental conditions.

F. Separation of Indium (III) from Other Elements

The adsorption of different cations on zirconium phosphosilicate under the experimental conditions was studied. One milligram of the metal ion along with its tracer was mixed with 1.0 mg of In (III) and the total volume of the mixture was made to 10 cm^3 by the addition of distilled water. The pH of the solution was adjusted to 4.0 and was added to 200 mg of zirconium phosphosilicate. The mixture was stirred for 5.0 minutes and centrifuged. The percentage adsorption of the metal ion was calculated by measuring the radioactivity (tracer technique). As expected the percentage adsorption of the cations was greater without carrier than with carrier (Table III). The interfering cations were suppressed either by masking them with suitable masking agents or washing the exchanger with an appropriate reagent. The adsorption of Pd(II), Ca(II), W(VI), Sn(II), K(I), Fe(III), and Fe(II) was decreased to less than 10% by increasing carrier up to 20 mg and that of Sb(III), Sb(V) and Sc(III) was decreased to less than 1 % by washing the exchanger with 50 mg of tartrate. The adsorption of Pt(IV) and Sm(III) was decreased to less than 1% by washing the exchanger with 10% NH_4Cl solution and 100 mg of fluoride respectively. The adsorption of Gd(III), La(III), Ag(I) and Fe(III) was decreased to less than 2% by masking with 100 mg of EDTA. The interference of Cs(I), Tl(I), Ba(II) and Rb(I) was decreased by precipitating them prior to adsorption. From Table-III, it is clear that many of the elements such as Nd(III), Zr(IV), Se(IV), Ir(III), Ir(IV), Au(III), Zn(II), Cu(II), As(V), As(III), Mo(VI), Sn(IV), Na(I), Tc(VII), Ce(IV), Co(II), Sr(II), Cr(VI), Hg(II), Os(VI), S(VI), Cd(II), Er(III), Mn(II), Te(IV) and Re(VII) did not interfere with the separation of In(III) by adsorbing it on zirconium phosphosilicate under the experimental conditions.

TABLE-III

Percentage Adsorption of Other Elements on Zirconium Phosphosilicate
(Amount of exchanger= 200 mg; pH= 4.0; total volume of solution= 10 cm³);

% Adsorption	Elements	
	Without carrier	With carrier
< 1.0	Sn ²⁺ , Sb ^{3+c} , Sb ^{5+c} , S ⁶⁺ , Ag ^{+b} , Sm ^{3+c} , Re ⁷⁺ , Fe ^{3+b} , Ba ^{2+b} , Sc ^{3+c} , Rb ^{+d} .	Gd ^{3+b} , Ir ³⁺ , Ir ⁴⁺ , Mo ⁶⁺ , Sn ⁴⁺ , Pt ^{4+c} , La ^{3+b} , Sb ^{3+c} , Sb ^{5+c} , Sc ^{3+c} , Re ⁷⁺ , Ba ^{2+d} , Rb ^{+d} , Sm ^{3+c} .
1-9.99	Nd ³⁺ , Zr ⁴⁺ , Ca ²⁺ , Se ²⁺ , Ir ³⁺ , Ir ⁴⁺ , Au ³⁺ , W ⁶⁺ , Zn ²⁺ , Cu ^{2+c} , Pd ²⁺ , As ⁵⁺ , As ³⁺ , Mo ⁶⁺ , Sn ⁴⁺ , Cs ^{+d} , K ⁺ , Na ⁺ , Pt ⁴⁺ , La ^{3+b} , Te ⁴⁺ , Ce ⁴⁺ , Co ²⁺ , Sr ²⁺ , Cr ³⁺ , Cr ⁶⁺ , Hg ²⁺ , Os ^{6+c} , Cd ²⁺ , Er ³⁺ , Mn ²⁺ , Te ⁴⁺ , Fe ³⁺ , Fe ²⁺ .	Zr ⁴⁺ , Ca ^{2+a} , Gd ^{3+b} , Se ^{4+b} , Au ³⁺ , W ^{6+a} , Zn ²⁺ , Cu ²⁺ , Pd ^{2+a} , As ⁵⁺ , As ³⁺ , Sn ^{2+d} , Cs ^{+d} , K ^{+a} , Na ⁺ , Ce ⁴⁺ , Co ²⁺ , Sr ²⁺ , Cr ³⁺ , Cr ⁶⁺ , Hg ²⁺ , Os ⁶⁺ , S ⁶⁺ , Cd ²⁺ , Mn ²⁺ , Te ⁴⁺ , Fe ^{3+a} , Fe ^{2+a} .
10-50	Gd ³⁺ , Cu ²⁺ , Sn ²⁺ , La ³⁺ , Sb ³⁺ , Sb ⁵⁺ , Os ⁶⁺ , Ag ⁺ , Sm ³⁺ , Ba ²⁺ , Sc ³⁺ , Rb ⁺ .	Gd ³⁺ , W ⁶⁺ , Pd ²⁺ , Sn ²⁺ , K ⁺ , Ca ²⁺ , Pt ⁴⁺ , Sb ³⁺ , Sb ⁵⁺ , S ⁶⁺ , Sm ³⁺ , Fe ³⁺ , Fe ²⁺ , Ba ²⁺ , Sc ³⁺ , Rb ⁺ .
> 50	Cs ⁺	Cs ⁺ , La ³⁺ .

a = Increased carrier

b = Masking with an appropriate reagent

c = Removed by washing the ion-exchanger d = Precipitated prior to adsorption

G. Distribution coefficient (Kd) values

The distribution co-efficient values (Kd) of different elements (1.0 mg each) in presence of 1.0 mg of In (III) carrier was evaluated. $K_d = (C_o / C_e - 1) \cdot A / m$, Where Kd is distribution co-efficient, Co is original concentration of solute in the solution, Ce is the concentration of solute in the solution at equilibrium, A is the total volume of the solution, m is the amount of adsorbent taken for batch equilibration. The Kd values of Ir(III), Re(VII), Ir(IV), Mo(VI), As(III), Sn(IV), Zr(IV), Cd(II), Co(II), As(V) and S(VI) were found to be less than 1.0, while that of Hg(II), Te(IV), Zn(II), Se(IV), Mn(II), Ce(III), Cr(VI), Na(I), Cu(II), Au(III), Cr(III), Sr(II), Os(VI), Fe(II), Pd(II), Ca(II), Fe(III), K(I) and W(VI) was found to be between 1.0 to 10.0. The Kd value of Pt(IV), Sb(III), Rb(I), Ba(II), Gd(III), Sc(III), Sb(V), Sn(II) and Sm(III) was found to be in the range of 11-40; whereas, the Kd value of La(III) and Cs(I) was found to be 91 and 154 respectively. The distribution co-efficient value of these elements was decreased to below 5.0 by either increasing the amount of carrier of a concerned element or washing the exchanger with water of pH 4.0 or by masking with a suitable masking agent. The Kd values reveal that the affinity of Cs (I), La (III) towards zirconium phosphosilicate is more as compared to other elements under experimental conditions.

The decontamination factor (D.F.) for various elements (with and without a carrier) was determined under the experimental conditions. The D.F. values of some elements were improved by increasing carrier, washing the exchanger with an appropriate reagent or by masking. It is observed that most of the elements have D.F. values to be better than 10².

CONCLUSIONS

From the above discussion, it is apparent that the method developed is highly selective and rapid for the separation of In (III). The method thus developed will be employed for the radiochemical separation of In (III) for its estimation by NAA. 200 mg of zirconium phosphosilicate is sufficient for maximum absorption (94.48%) of 1.0 mg of In (III) at pH 4.0 and contact time of 5.0 minutes.

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