

Column Chromatographic Separation of Cadmium and Mercury from other elements on Dowex 21 K (Cl⁻) resin

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Abstract : The ion exchange chromatographic separations were carried out for Cadmium and Mercury using Dowex 21 K (Cl⁻) using butyrate as complexing agent. Various metal ions were studied in comparison to differentiate the behaviour of the metal ions. The results and discussion of the study are presented in the form of paper below.

Key Words : Chromatographic Separation / Dowex 21 K (Cl⁻) / Distribution Coefficients/ Separation

The sorption and separation of metal carboxylate ions¹⁻⁴ by anion exchange resins has been studied in various experimental conditions The resin Dowex 21 K (Cl⁻) was obtained from Dow Chemical Company, Michigan, U.S.A. . It was treated and washed with water to achieve swelling. Most of the metal ion solutions (0.05 M) were prepared in glass distilled water from chlorides or nitrates.

The batch equilibrium method was to measure the distribution coefficients. Each equilibrium experiment was performed in 100 ml conical flask containing 54 ml of mixture of 4 ml of 0.05 M metal ion solution and 50 ml of the solvent system. To this mixture 1 g resin was added and the mixture was equilibrated for 24 h. The metal ion content was determined by suitable volumetric method. The distribution coefficients (KD) was determined by using the formula^{2,5,6} .

$$K_D = \frac{\text{m mol of metal ion on resin / g of dry resin}}{\text{m mol of metal ion in solution / total volume of solution}}$$

Separation of mixture was carried out by feeding 10 ml metal ion mixture on the column of 20 cm height and 1 cm inner diameter. The metal ions were eluted by suitable eluting agents. The effluents were collected in number of fractions of 10 ml each and estimated volumetrically⁷ . The distribution coefficients of Mg, Ca, Th, Hg, Pb, Mn Co, Cu, Ni, and Al (Table 1) were determined at 0, 20, 40 ,60, and 80 percentage of acetone and at 0.5 M n-Butyric Acid Media.

The values of K_D at this concentration are mentioned in the table no. 1 given below.

Table 1 –Distribution coefficient values of different metal ions in Aqueous-Acetone- 0.5 M n-Butyric Acid Media on Dowex 21 K (Cl⁻)

Metal Ion	Acetone Concentration , % (v/v)				
	0	20	40	60	80
Mg(II)	1.42	NA	NA	NA	1.72
Ca (II)	NA	NA	NA	20.4	18.2
Th(II)	3.62	5.08	2.05	1.75	9.66
Cd(II)	12.40	68.89	183.6	1015.2	1728.0
Hg(II)	967.0	1350.0	1550.5	5562.0	TA
Pb(II)	NA	NA	0.55	37.24	568.58
Mn (II)	NA	NA	NA	NA	4.85
Co(II)	1.08	NA	1.36	NA	1.92
Cu(II)	NA	NA	NA	NA	NA
Ni(II)	NA	0.72	0.36	1.86	1.86
Al(II)	3.62	5.08	2.05	1.75	9.66

NA = No Adsorption , TA = Total Adsorption .

The distribution coefficients of Mg, Ca, Th ,Mn, Co ,Cu Ni and Al are low at all percentages of acetone. The distribution coefficient of Hg indicates total adsorption at 80 percent concentrations of acetone .This indicates a very strong tendency of complete adsorption of Hg. Similarly Cd shows very high values of adsorption at 60 and 80 percentage of acetone. Therefore it can be concluded that Cd and Hg are completely held by the resin at higher concentrations of acetone⁷ .

Based on these studies the separation of Cd and Hg from other metal ions was undertaken. The binary separation means separation of two metal ions from the solution and tertiary means separation of three metal ions from each other. Although more than 100 separation were achieved by making different combinations, the present paper records only a few of these separations.

Separation of Cd and Hg from Mg / Ca / Pb / Mn / Co / Cu /Ni :

As indicated by distribution coefficient values, Cd and Hg are completely adsorbed on the resin and they shows very high adsorption . In comparison to this Mg / Ca / Pb / Mn / Co / Cu /Ni show very low values of adsorption. All these metal ions are separated form the mixture by passing 60 % acetone – 0.5 M butyric acid and 20 % Acetone 0.5 M butyric acid at as eluent. Cd was later eluted from the column by 1 M chloroacetic acid.(Table no.2). Mercury was eluted with 2 M trichloroacetic acid.

Table 2: Quantitative separation of the Synthetic Binary Mixture
 (First metal ion is eluted first, while the second is retained.)

Mixture	Metal ion eluted	Eluting agent	m moles taken	m moles found
Mg (II) + Cd (II)	Mg (II)	a	0.241	0.232
	Cd (II)	b	0.247	0.232
Ca (II) + Cd (II)	Ca (II)	a	0.244	0.242
	Cd (II)	b	0.247	0.232
Pb(II) + Cd (II)	Pb(II)	a	0.249	0.245
	Cd (II)	b	0.247	0.232
Mn (II) + Cd (II)	Mn (II)	a	0.244	0.233
	Cd (II)	b	0.247	0.232
Co (II) + Cd (II)	Co (II)	a	0.247	0.246
	Cd (II)	b	0.247	0.232
Cu (II) + Cd (II)	Cu (II)	a	0.244	0.243
	Cd (II)	b	0.247	0.232
Ni (II) + Cd (II)	Ni (II)	a	0.244	0.241
	Cd (II)	b	0.247	0.232
Th (II) + Cd (II)	Th (II)	a	0.250	0.246
	Cd (II)	b	0.247	0.232
Mg(II) + Hg (II)	Mg(II)	c	0.241	0.240
	Hg (II)	d	0.192	0.191
Ca(II) + Hg (II)	Ca(II)	c	0.242	0.235
	Hg (II)	d	0.192	0.191
Cd(II) + Hg (II)	Cd(II)	c	0.250	0.249
	Hg (II)	d	0.192	0.191
Pb(II) + Hg (II)	Pb(II)	c	0.248	0.247
	Hg (II)	d	0.192	0.191
Mn(II) + Hg (II)	Mn(II)	c	0.252	0.23
	Hg (II)	d	0.192	0.191
Co(II) + Hg (II)	Co(II)	c	0.271	0.270
	Hg (II)	d	0.192	0.191
Cu(II) + Hg (II)	Cu(II)	c	0.224	0.222
	Hg (II)	d	0.192	0.191
Ni(II) + Hg (II)	Ni(II)	c	0.244	0.242
	Hg (II)	d	0.192	0.191

a = 60 % Acetone - 0.5 M Butyric Acid
 b = 1 M Chloroacetic Acid
 c = 20 % Acetone – 0.5 M Butyric Acid
 d = 2 M Chloroacetic Acid

The various ions were eluted and the fractions of 5-10 ml were collected and estimated. It was generally observed that elution starts quickly in the first 5-19 ml (Break Through Volume BTV). The elution is maximum at 10 -40 ml (Volume at Elution Peak VEP) and the elution was complete at 60-90 ml (Terminal Elution Volume TEV) as the flow rate in most of the separations was maintained 1 ml/ min. The separation was stated within 5 -10 min, reached to maximum in 30-40 min and became complete in 1 h to 1.5 h.

The recovery of each constituent metal ion in the mixtures was more than 90 % with relative standard deviation (RSD) 2.5 %. The elution curves of the separation of the mixtures were distinct and Gaussian.

REFERENCES :

[1] Kulkarni, S.V., Soman, S.K. and Joshi, S.S.(1983) J. Indian Chem. Soc.. 60.:779
 [2] Korkisch, J.And Urubay, S. (1994), Talenta 11:721
 [3] Pyantnitskii,I.V.,(1998)Izu.Vyshh.Uchuben.Zaved. Khim.Teknol 6:20
 [4] Kulkarni ,S.V. And Rathod, A.A.(1992) J. Ind. Council Chem . 8(2) :6
 [5] Kulkarni ,S.V. And Rathod, A.A. (1991) Talenta 23: 534
 [6] Kulkarni ,S.V. And Rathod, A.A. (1990) J.American Chemical Society , IV: 78-83
 [7] Vogel ,A.I.(1961) “ A Text Book of Quantitative Inorganic Analysis” , Longmans Green , London, p 319.