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# Studies on the sorption of praseodymium (III), holmium (III) and cobalt (II) from nitrate medium using TVEX–PHOR resin

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#### Abstract

The use of TVEX–PHOR resin for the sorption of praseodymium (III), holmium (III) and cobalt (II) from nitrate medium was carried out using batch and column techniques. Various parameters affecting the uptake of these metal ions such as v/m ratio, pH and the metal ion concentration were separately studied. Effect of temperature on the equilibrium distribution values has been studied to evaluate the changes in standard thermodynamic quantities. Experimental results of the investigated metal ions were found to fit to Freundlich isotherm model over the entire studied concentration range. Selectivity sequence of the resin for these metals is Ho > Pr > Co. The recovery of the investigated metals from the loaded resin is preformed with 0.1 M sulphuric acid.

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## 1. Introduction

Solid phase extraction is one of the techniques which found wide application in the nuclear processing industry, particularly in separation of fission products. Many types of ion exchange resins and chelates such as polystyrene [1–4], poly vinyl resin [5] and polyacrylate [6] were used for solid phase extraction of metal ions.

Lanthanide elements have gained a great attention in the last few decades owing to their unique properties and wide range of applications that utilize these elements in huge quantities. These elements and their compounds have found a variety of applications especially in metallurgy, ceramic industry, and nuclear fuel control [7,8]. Gadolinium oxide, for example, one of its largest uses in nuclear power reactors is as shielding and fluxing devices, while praseodymium is used in the production of atomic batteries [9]. The separation of lanthanides in different matrices using solid phase extraction has been performed by several authors [10–12].

Cobalt, which is known as a corrosion product in cooling water system in nuclear power plants [13], is also found in

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radioactive waste solutions resulting from activities related to nuclear fuel cycle [14], and from medical applications.

Several studies dealing with sorption and separation of cobalt with solvent impregnated resins (SIR) have been carried out. Isshiki and Nakayama [15] examined the selective concentration of trace amounts of cobalt in sea water using 14 different materials; such as 2-(2-thiozolylazo)-*p*-cresol and 2-(2-thiozolylazo) resorcinol, and two XAD resins (styrene polymer and acrylate polymer) types. They concluded that, the combination of TAR and XAD-4 is the most preferable choice. Amberlite XAD-2 loaded with 2-(2-thiozolylazo)-*p*-cresol (TAC) [16] is used for separation and preconcentration of Cobalt. The sorption of cobalt from nitric acid solution by bis (2,4,4-trimethyl pentyl) dithiophosphinic acid (CYANEX 301) loaded cellulose sorbent material (Egy-Sorbs) as an inert supporting material has been examined through batch kinetics and equilibrium studies [17].

TVEX solid phase has been used for the uptake and separation of many elements. Kremliakova et al. [18] studied the separation of tetravalent plutonium from trivalent actinides and lanthanides from strong nitric acid solution using TVEX containing TBP, HDEHP and TOA. Popik and Zajtsev [19] examined the extraction and separation of palladium and rare earth metals using TVEX–PHOR resin. TVEX–TBP [20] has been examined for extraction of niobium. The authors have reported that, niobium is extracted from hydrochloric and sulphuric aqueous media at 7

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and 9 mol/l solutions niobium by both TBP and TVEX–TBP as:  $[H_3O(m-1)H_2O \cdot nTBP]^+[NbOCl_4]^-$  anion complex in which the metal is not bounded with TBP directly.

El-Nadi and Daoud [21] used TVEX–PHOR resin for separation of uranium and thorium from sulphuric acid medium, and reported the affinity of the resin towards the extraction of uranium than thorium; maximum separation of both ions is obtained from 3 M sulphuric acid.

In this paper, TVEX–PHOR resin is used for the extraction and separation of praseodymium (III), holmium (III) and cobalt (II) from nitrate medium. The parameters affecting the sorption of metals such as initial metal concentration, contact time and pH are investigated, desorption of metal ions is also studied and discussed. The column investigations are also carried out to throw light on the separation of lanthanides and Cobalt from nitrate medium.

# 2. Experimental

#### 2.1. Chemicals and reagents

All chemicals and reagents used were of analytical grade. Praseodymium and holmium oxides were Fluka products. Cobalt and sodium nitrate were from BDH. Arsenazo (III) was supplied by Merck. The TVEX–PHOR resin was kindly supplied by Pridneoprovsky chemical plant, Ukraine. The specifications given in the technical sheet show that, the resin consists of macroporous copolymer of styrene and divinyl benzene where the functional group is octyl, heptyl, pentyl phosphine oxide. The extraction fraction of total mass adsorbent is  $50 \pm 1\%$ . Swelled in water adsorbent specific volume is 1-1.1 ml/g and its particle size is 0.6-1.6 mm. The total surface area of the TVEX–PHOR resin was measured using standard volumetric method by nitrogen gas adsorption at 77 K and application of the BET-equation. The values of the specific surface area and degree of porosity for the resin were  $860 \text{ m}^2/\text{g}$  and 68%, respectively.

# 2.2. Procedure

Praseodymium and holmium solutions were prepared by dissolution of certain weights of their oxides in nitric acid, heated to dryness, washed several times with double distilled water and dissolved in 0.1 M sodium nitrate to obtain solutions containing 0.5 g/l for each metal at pH  $3.5 \pm 0.1$ . Praseodymium and holmium concentrations were determined spectrophotometrically by measuring their maximum absorbance at 578 nm by Arsenazo (III) method [22] using a Shimadzu double beam recording Spectrophotometer model 160A. Cobalt concentration in the aqueous nitrate solution was determined by Z-8100 Polarized Zeeman, Atomic Absorption Spectrophotometer.

Batch sorption experiments were performed by shaking 0.05 g TVEX–PHOR with 5 ml of the metal solution in a thermostated shaker bath adjusted at 25 °C. The amount of metal uptake was calculated by the difference between the equilibrium concentration and the initial concentration. The amount of metal retained in the resin  $q_e$  (mg/g), was calculated using



Fig. 1. Basic experimental set-up for column chromatography.

the relation:

$$q_{\rm e} = (C_0 - C_{\rm e}) \left[ \frac{v}{m} \right] \tag{1}$$

where  $C_0$  and  $C_e$  are the initial and equilibrium concentrations (mg/l) of metal ions in solution, respectively, v the volume (l) and m is the weight (g) of the adsorbent.

The distribution coefficient  $(K_d)$  of metal ions between the aqueous phase and the resin phase is calculated from the relation:

$$K_{\rm d} = \left[\frac{C_0 - C_{\rm e}}{C_{\rm e}}\right] \left[\frac{v}{m}\right] \tag{2}$$

$$K_{\rm d} = \frac{q_{\rm e}}{C_{\rm e}} \tag{3}$$

#### 2.3. Preparation of chromatographic column

Perspex chromatographic column, Fig. 1, with 1 cm inside diameter and 7 cm long was used. The column was filled with 2 g with a bed height of 5 cm resin using a pump. A solution of 100 ml of loading solution containing 100 ppm of holmium and cobalt was passed through TVEX–PHOR resin with a flow rates 0.5, 1, and 1.5 ml/min. After elution, 3 ml fraction volumes were collected for analysis at different time intervals. All experiments were carried out at room temperature ( $25 \pm 1^{\circ}$ C).

# 3. Results and discussion

#### 3.1. Kinetic studies

The sorption kinetic of praseodymium (III), holmium (III), and cobalt (II) on the TVEX–PHOR exchange resin were studied as a function of contact time at different metal ion concentrations (100, 300 and 500 mg/l) in the range 5–120 min. As shown in (Fig. 2a–c), the sorption of metal ions increased with time and attained equilibrium after 60 min for praseodymium (III), 90 min for holmium (III) and 30 min for cobalt (II) for all the initial metal ion concentrations used. Cobalt ion did not adsorb onto TVEX–PHOR at 500 mg/l concentration and the



Fig. 2. (a) Effect of contact time and initial concentration of Pr(III) on its adsorption on TVEX–PHOR, (b) effect of contact time and initial concentration of Ho(III) on its adsorption on TVEX–PHOR and (c) effect of contact time and initial concentration of Co(II) on its adsorption on TVEX–PHOR.

percentage of adsorption was minimal for the concentrations of 100 and 300 mg/l. This indicates that the equilibrium time is independent of the initial metal ion concentration.

To describe the changes in the sorption of the studied ions with time, several kinetic models were tested. The rate constant of each metal ion removal from the aqueous solution by TVEX–PHOR was determined using pseudo-first order and



Fig. 3. Lagergren plots for the adsorption of Pr(III), Ho(III), and Co(II) on TVEX–PHOR resin.

pseudo-second order rate models. The Lagergren first order expression [23,24] is given by:

$$\log\left(q_{\rm e} - q_t\right) = \log q_{\rm e} - K_{\rm ad}\left(\frac{t}{2.303}\right) \tag{4}$$

where  $q_e$  and  $q_t$  are the amounts of metal ion sorbed onto TVEX–PHOR (mg/g) at equilibrium and at time *t*, respectively, and  $K_{ad}$  is the rate constant of first order adsorption (min<sup>-1</sup>). Linear plot of  $(q_e - q_t)$  versus *t*, Fig. 3, showed that the sorption of ions followed the Lagergren equation over the entire range of contact time explored. The first order rate constants  $K_{ad}$  were evaluated from the slope of linear plots obtained and found to be  $5.4 \times 10^{-2}$ ,  $8.3 \times 10^{-2}$  and  $3.1 \times 10^{-2}$  min<sup>-1</sup> for praseodymium (III), holmium (III), and cobalt (II), respectively for a fixed concentration of 100 mg/l for each metal ion. The correlation coefficient ( $R^2$ ) suggests a strong relationship between the parameters and also explains that the process of sorption of each metal ion follows pseudo-first order kinetics.

# 3.2. Effect of v/m ratio

The effect of changing v/m (ml/g) ratio on the adsorption of praseodymium (III), holmium (III) and cobalt (II) on TVEX–PHOR was studied in the range 25–300 to evaluate the optimum resin weight per unit solution volume that could be used for a high adsorption capability. As shown in Fig. 4, the adsorption percent of praseodymium (III) and holmium (III) increases as v/m decreases from 300 to 100 ml/g and then remains constant with further decrease of v/m up to 25 ml/g. Thus, the optimum v/m ratio was kept at 100 ml/g during all praseodymium (III) and holmium (III) experiments while cobalt (II) was not adsorbed at v/m higher than 50 ml/g.

## 3.3. Effect of pH

In order to optimize the pH for maximum adsorption efficiency, the adsorption was studied in the pH range 2–7 and the results are depicted in Fig. 5. It was observed that the adsorption of holmium (III) and cobalt (II) on TVEX–PHOR is low in the acidic solutions and increases by increasing the pH values. The



Fig. 4. Effect of v/m ratio on the adsorption of Pr(III), Ho(III), and Co(II) on TVEX–PHOR resin.

maximum adsorption of praseodymium (III) and holmium (III) was attained at nearly 3.5 while that of cobalt (II) was attained at nearly 5.5.

## 3.4. Effect of temperature

The effect of temperature on the adsorption of praseodymium (III), holmium (III) and cobalt (II) on the TVEX–PHOR resin in aqueous nitrate medium have been examined at different temperatures in the range 15–45 °C. It was observed that the sorption of these metal ions slightly increases with increasing temperature. The Van't Hoff equation [25] given below, can be used to calculate the enthalpy changes associated with the sorption of these metal ions:

$$\log K_{\rm d} = -\left(\frac{\Delta H}{2.303R}\right)\frac{1}{T} + C \tag{5}$$

where *R* is the universal gas constant,  $K_d$  the distribution coefficient, *T* the absolute temperature and *C* is a constant.

The plots of  $\log K_d$  versus 1/T for the sorption of praseodymium, holmium and cobalt ions are shown in Fig. 6. The estimated values of  $\Delta H^{\circ}$  are given in Table 1. The posi-



Fig. 5. Effect of pH on the adsorption of 100 mg/l Pr(III), Ho(III), and Co(II) on TVEX–PHOR resin.



Fig. 6. Effect of temperature on the adsorption of Pr(III), Ho(III), and Co(II) from nitrate medium (initial concentration 100 ppm and v/m = 100 for Pr and Ho and v/m = 50 for Co).

tive values of  $\Delta H^{\circ}$  indicate the endothermic type of the sorption process. Also, the higher  $\Delta H^{\circ}$  values for holmium than praseodymium and cobalt give evidence that holmium ion is more binding to the surface of sorbent than the other ions.

The Gibbs free energy change,  $\Delta G^{\circ}$ , is the fundamental criterion of spontaneity, reaction occurring spontaneously at a given temperature if it is a negative value. The free energy change  $\Delta G^{\circ}$  and the entropy change  $\Delta S^{\circ}$ , for the sorption processes are calculated using the following equations:

$$\Delta G = \Delta G^\circ + RT \ln K$$

At equilibrium,  $\Delta G = 0$ . Then,

$$\Delta G^{\circ} = -RT \ln K \tag{6}$$

$$\Delta G^{\circ} = \Delta H^{\circ} - T \,\Delta S^{\circ} \tag{7}$$

The calculated values of  $\Delta G^{\circ}$  and  $\Delta S^{\circ}$  for the studied metal ions are shown in Table 1. The negative  $\Delta G^{\circ}$  indicates a spontaneous nature of praseodymium (III), holmium (III) (-20 kJ/mol) are generally smaller than that of chemical adsorption (higher than -80 kJ/mol) [25].

As shown in Table 1, the magnitude of adsorption free energy  $(\Delta G^{\circ})$  ranging from -10.5 to -15.4 kJ/mol, suggests that the adsorption can be considered as a physical one.

The positive  $\Delta S^{\circ}$  values indicate an increase in the randomness of the investigated ion species due to the disruption of the ion-water and water-water interactions of the hydrated metal.

Table 1 Thermodynamic parameters for the sorption of Pr(III), Ho(III), and Co(II) from nitrate medium onto TVEX–PHOR resin

Metal ions	$\Delta H^{\circ} (\mathrm{kJ}\mathrm{mol}^{-1})$	$\Delta G^{\circ} (\text{kJ mol}^{-1})$	$\Delta S^{\circ} (\text{kJ mol}^{-1})$
Pr(III)	$15.89 \pm 0.11$	$-14.25 \pm 0.05$	$101.30 \pm 0.35$
Ho(III)	$2.7 \pm 0.13$	$-15.4 \pm 0.04$	$60.70\pm0.55$
Co(II)	$7.28\pm0.16$	$-10.5\pm0.27$	$59.66 \pm 1.44$



Fig. 7. Frenudlich isotherm for the sorption of Pr(III), Ho(III), and Co(II) onto TVEX–PHOR resin.

#### 3.5. Sorption isotherm

The experimental results obtained for the adsorption of praseodymium (III), holmium (III) and cobalt (II) on TVEX–PHOR resin at room temperature  $25 \pm 1$  °C under the optimum conditions of contact time and v/m ratio were found to obey Freundlich adsorption isotherm [26]. The Freundlich equation may be written as:

$$q_{\rm e} = K_{\rm f} C_{\rm e}^{1/n} \tag{8}$$

$$\log q_{\rm e} = \log K_{\rm f} + \frac{1}{n} \log C_{\rm e} \tag{9}$$

where  $K_{\rm f}$  and *n* are the Freundlich constants, which represent the adsorption capacity and intensity of adsorption, respectively. The Freundlich adsorption isotherm represents the relationship between the amount of metal adsorbed per unit mass of adsorbent  $(q_e)$  and the concentration of the metal in solution at equilibrium ( $C_e$ ). The plot of log  $q_e$  (mg/g) versus log  $C_e$  (g/l) for various initial concentrations was found to be linear Fig. 7, indicating the applicability of the classical adsorption isotherm to this adsorbate-adsorbent system. The fitting of the data to the Freundlish isotherm suggests that, sorption process is not restricted to one specific class of sites and assumes surface heterogeneity [27]. From the slope and intercept of the plots, the adsorption capacities  $(K_f)$  and adsorption intensities (n) for praseodymium (III), holmium (III) and cobalt (II) ions are calculated and listed in Table 2. The slope of the Freundlish isotherm for all metal ions is less than 1, values of 1 > n > 10 show favorable adsorption of metal on TVEX-PHOR resin. The sorption capacity values which calculated using relation (1) is considerably differ-

Table 2

Parameters of Freundlich isotherm for sorption of Pr(III), Ho(III), and Co(II) ions onto TVEX–PHOR resin

Metal ions	Parameters for Freundlich model			Apparent capacity
	$\overline{K_{\rm f}~({\rm mg/g})}$	n	$R^2$	$q_{\rm e}  ({\rm mg/g})$
Pr(III)	1.3	2.5	0.999	49.0
Ho(III)	1.4	2.0	0.999	50.4
Co(II)	0.5	3.3	0.999	8.7



Fig. 8. Langmuir isotherm for the sorption of Pr(III), Ho(III), and Co(II) onto TVEX–PHOR resin.

ent from that deduced from Freundlish isotherm, Table 2. This difference is due to that  $K_f$  is the relative adsorption capacity indicative to the selectivity order for the investigated metal ions. Higher values for  $K_f$  indicate higher affinity for holmium (III), praseodymium (III), and cobalt (II). The apparent capacity ( $q_e$ ) is a quantitative value for the capacity power for the TVEX–PHOR resin and follows the same order.

The Langmuir model was tested in its linear form:

$$\frac{C_{\rm e}}{q_{\rm e}} = \frac{1}{Q^0 b} + \left(\frac{1}{Q^0}\right) C_{\rm e} \tag{10}$$

where  $q_e$  is the amount sorbed per unit weight of adsorbent (mg/g),  $C_e$  the equilibrium concentration of the solute in the bulk solution (mg/l),  $Q^0$  the monolayer adsorption capacity (mg/g) and *b* is the constant related to the free energy of adsorption (sorption energy). The plots of  $C_e/q_e$  against  $C_e$  for the investigated metal ions sorption are represented in Fig. 8. The relationships are not linear and the values of correlation coefficient ( $R^2$ ), suggests that, the Langmuir model does not fit with the obtained results.

## 3.6. Desorption studies

The recovery of praseodymium (III) and holmium (III) from loaded TVEX–PHOR resin was studied using different concentrations of hydrochloric, nitric, and sulphuric acids in the range 0.1–2 M. The results obtained are listed in Table 3. The stripping percentage of both praseodymium (III) and holmium (III) using nitric acid was very poor in the studied concentration range.

The increase in hydrochloric acid concentration in the investigated range, showed an increase in the stripping percent of praseodymium (III) and holmium (III). The stripping of praseodymium (III) reached 56% and holmium (III) 93.5% at 0.5 M and remains constant with further increase in acid concentrations.

On the other hand, the increase in sulphuric acid concentration decreased the stripping of praseodymium (III) and holmium (III), Table 3. 0.1 M sulphuric acid stripped holmium

Table 3	
Stripping of Pr (III) and Ho(III) from loaded TVEX-PHOR resin using differen	nt
acids	

Stripping agent	Stripping percent (%)		
	Pr(III)	Ho(III)	
HNO <sub>3</sub> (M)			
0.1	0.5	-	
0.3	1.0	-	
0.5	1.5	5.0	
1.0	1.7	6.0	
2.0	2.5	11.0	
HCl (M)			
0.1	15	15.5	
0.3	61	40	
0.5	56	93.5	
1.0	56	93.5	
2.0	56.5	93.5	
$H_2SO_4(M)$			
0.1	9.2	70.0	
0.3	7.2	34.5	
0.5	3.4	6.7	
1.0	1.8	3.5	
2.0	-	-	

with 70% while it stripped praseodymium (III) with 9% only. This indicates that the use of 0.1 M sulphuric acid as stripping agent could give a better separation between holmium (III) and praseodymium (III).

# 3.7. Column investigations

A chromatographic column was constructed as given in the experimental section. The breakthrough curve was first studied in terms of aforementioned effluent composition, using a flow rates 0.5, 1.0, and 1.5 ml/min. To this column, 100 ml of loading solution containing 100 ppm of holmium (III) and cobalt (II) was percolated through the column with different flow rates, Fig. 9. The breakthrough curves obtained with TVEX–PHOR at different flow rates show that, by decreasing flow rate from 1.5 to 1.0 ml and then to 0.5 ml/min, the number of column volumes



Fig. 9. Ho(III) and Co(II) breakthrough curves with TVEX–PHOR from 0.1 M nitrate medium at different flow rates.

to bring a 50% breakthrough increased from 7 to 10 ml and then to 19 ml, respectively, for holmium. In the case of cobalt (II), due to its low adsorption percent, the bed volumes increased from 5 to 7 ml and then to 10 ml for 90% breakthrough. The column results show a higher capacity of TVEX–PHOR resin for the sorption of holmium (III) and very low capacity for the sorption of cobalt (II), which agrees with the results obtained in batch studies. The elution of holmium (III) from the column was obtained with 0.1 M sulphuric acid.

### 4. Conclusions

- The results of praseodymium (III), holmium (III), and cobalt (II) ions sorption reported that, 85% sorption was achieved for holmium (III), 75% for praseodymium (III), and 12% for cobalt (II). This enables the possibility of separation of cobalt (II) from the investigated lanthanide elements.
- The sorption of the investigated metal ions increases by increasing the contact time and decreasing by increasing v/m ratio and metal ion concentration. The sorption process is endothermic in nature.
- The equilibrium isotherms for sorption of the investigated metal ions have been modeled successfully using the Freundlich isotherm.

Elution studies show that 70% recovery was achieved for holmium (III) by 0.1 M sulphuric acid, but in the case of praseodymium (III) a recovery value of 9% only was possible, which could enable the separation of holmium (III) from praseodymium (III)

• The results of the column investigations were found to be similar to those of the batch investigations. The breakthrough capacity of holmium (III) is higher than that of cobalt (II) and the change in flow rate has a little effect on the shape and the breakthrough capacity of the curve.

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