Selective Adsorption of Europium on Activated Charcoal from Aqueous Solutions

M. Saleem*, M. Afzal Department of Chemistry, Quaid-i-Azam University, Islamabad, Pakistan.

Riaz Qadeer and Javed Hanif Pakistan Institute of Nuclear Science and Technology, P.O. Box 1356, Islamabad, Pakistan.

(Received 8 May 1992; accepted 25 November 1992)

ABSTRACT: The adsorption of europium on activated charcoal has been studied as a function of shaking time, pH, concentration of adsorbate and temperature. The adsorption of europium obeys the Langmuir and Dubinin-Radushkevich isotherms. Thermodynamic quantities such as $\Delta H^0$ and $\Delta S^0$ have been calculated from the slopes and intercepts of plots of $\ln K_D$ versus $1/T$. The influence of different cations and anions on europium adsorption has been examined. The adsorption of other metal ions on activated charcoal has been investigated under specified conditions to check their selectivity for europium. As a result, europium has been successfully removed from a mixture containing Ba, Sr, Ru, Cs, Cr, Nd, Pr and In. Approximately 96% of the europium adsorbed on the activated charcoal was recovered using 45 ml of 3 M HNO$_3$. X-ray fluorescence spectrometry was used for measuring the europium concentration.

INTRODUCTION

The adsorption of europium on solids is important from the viewpoint of purification, environmental and radioactive waste disposal. Some workers have studied the adsorption of europium on tungsten (Loginov and Mittsev 1980; Kolaczkiewiez and Bauer 1985), on calcium, zinc and cadmium oxalates (Massart 1969; Pazukhin 1981; Pencheva et al. 1980, 1984), on manganese dioxide (Cvjeticanin et al. 1982; Hasany and Chaudhary 1986), on barium and strontium sulphate (Ishina et al. 1971; Grebenshchikova et al. 1971, 1972; Persin 1983), on thorium oxalate deposit (Grebenschikova et al. 1970), on silver bromide and silver iodide precipitates (Herak 1969; Hus and Herak 1976), on plastics and glass (Ichikawa and Sato 1969), on silica gel (Akatsu et al. 1965; Caletka and Zaitseva 1969), on alumina (Shiao et al. 1980) and on iron oxide (Music et al. 1979). No data are available for its adsorption on activated charcoal. We have previously used activated charcoal for the preconcentration and separation of uranium (Saleem et al. 1992), thorium (Qadeer et al. 1992a), gadolinium (Qadeer et al. 1992b) and strontium (Qadeer et
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al. 1992c). The present work describes our investigation of the adsorption of europium on activated charcoal with a similar purpose in mind.

EXPERIMENTAL

Chemicals and reagents

The chemicals used in this study were activated charcoal (BDH; Item No. 33032), europium nitrate (Rare Earth Product, 99.999%), cerium nitrate (Rare Earth Product, 99.999%), lithium nitrate (Fluka, Item No. 62574), cobalt nitrate (Fluka, Item No. 60833), zinc nitrate (Fluka, Item No. 96482), chromium nitrate (Fluka, Item No. 27080), cobalt sodium acetate (Fluka, Item No. 71179), sodium thiosulphate (Fluka, Item No. 72049), sodium iodide (Fluka, Item No. 71710), sodium chloride (Fluka, Item No. 71378), sodium nitrate (Fluka, Item No. 71755) and ethylenediamine tetra-acetic acid (Fluka, Item No. 03610). Buffer solutions of different pH supplied by Fluka were used for the pH studies.

Instruments

A Siemens wavelength dispersive X-ray fluorescence (WDXRF) spectrometer, model SRS-200, was used with the following attachments: Cr X-ray tube; Soller slit with an angular divergence of 0.15°; and an LiF(100) crystal. An NaI(Tl) scintillation counter, linked through an universal interface LC-200 to a PDP-11/04 computer, was used for measuring the europium concentration. The pH measurements were undertaken using a Metrohm digital pH meter, model 605. An Edmund Buhler SM25 shaker was used for shaking the sample solutions at a constant speed of 150 rev/min.

Procedure

Adsorption measurements were carried out via a batch technique at room temperature (21±1°C) except where otherwise specified. Known amounts of activated charcoal in 250 ml reagent bottles containing 10 ml of europium solution were shaken for a given time period. The solutions were then filtered and the concentration of europium before and after shaking measured by means of the WDXRF spectrometer. The sample solutions contained in a 0.01 mm thick-walled polyethylene bottle were inserted in the spectrometer (Saleem et al. 1990). The distribution coefficient ($K_D$) and percentage adsorption (%) were computed using the following equations:

$$K_D = \frac{C_1}{C_2} \quad (1)$$

and

$$\% \text{ Adsorption} = \frac{100K_D}{K_D + \frac{V}{M}} \quad (2)$$
where $C_1$ is the amount of Eu$^{III}$ ions adsorbed per gram of solid and $C_2$ is the concentration of Eu$^{III}$ ions per ml of aqueous solution. In equation (2), $V$ is the volume of the solution and $M$ the amount of adsorbent.

RESULTS AND DISCUSSION

The adsorption of europium on activated charcoal has been studied as a function of shaking time. For such studies, 10 ml of the europium-containing solution (2000 μg/ml) was shaken with 100 mg of activated charcoal for different intervals of time over the range 2-60 min. Figure 1 shows the variation in percentage adsorption and in the distribution coefficient ($K_D$) with shaking time. Adsorption increases with an increase in shaking time and assumes a constant value at ca. 30 min when adsorption equilibrium was attained. For this reason, a shaking time of 30 min was selected for all further studies.

![Figure 1. Adsorption of europium on activated charcoal as a function of shaking time: curve (a), % adsorption; curve (b), $K_D$.](image)

The Weber and Morris equation (Weber and Morris 1963) was applied for time-dependent studies. This equation may be written as:

$$Q_t = k'(t)^{1/2}$$

where $Q_t$ is the amount of adsorbate at time $t$ and $k'$ is the intraparticle transport rate constant. The plot of $Q_t$ versus $t^{1/2}$ depicted in Figure 2(a) deviates from linearity, indicating that no intraparticle transport mechanism was involved in the process. The reaction
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order for the adsorption of europium on activated charcoal has been evaluated using the Langergren equation (Langergren 1898; Bhutani et al. 1991) in the form:

\[ \log(a_e - a_t) = \log a_e - \frac{k}{2.303} \ t \]  

(4)

where \( a_e \) is the amount of europium ions adsorbed at equilibrium, \( a_t \) the amount of europium adsorbed after a time \( t \) and \( k \) is the rate constant. The straight line obtained by plotting \( \log(a_e - a_t) \) against \( t \) [Figure 2(b)] indicates that the adsorption of europium corresponds to a first order process. The value of the rate constant, \( k \), obtained from the slope of the curve was 0.239/min.

![Figure 2. (a) Plots of the Weber-Morris, (b) Lagergren and (c) Bangham equations applied to the adsorption of europium on to activated charcoal.](image)

The Bangham equation (Aharoni et al. 1979) has also been applied to the above process in the form:

\[ \log \log \left( \frac{\phi_0}{\phi_0 - q_t W} \right) = \log \left( \frac{K_0 W}{2.303 V} \right) + \alpha \log \ t \]  

(5)

where \( \phi_0 \) is the amount of Eu\textsc{iii} ions present in the solution, \( V \) the volume of the solution, \( W \) the weight of activated charcoal employed and \( q_t \) the amount of europium ions adsorbed at time \( t \); \( K_0 \) is a proportionality constant and \( \alpha \) is a constant whose value should be less than unity. A straight line may be obtained on plotting \( \log \log(\phi_0/(\phi_0 - q_t W)) \) against \( \log \ t \) [Figure 2(c)], indicating that the system acts in accordance with the
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Bangham equation. The values of $\alpha$ and $K_0$ calculated from the slope and intercept of the plot were 0.087 and 73.13, respectively.

![Graph showing adsorption and distribution coefficient (K_D) vs pH](image)

Figure 3. The influence of pH on the adsorption of europium on to activated charcoal: curve (a), % adsorption; curve (b), K_D.

Figure 3 shows the influence of pH on the adsorption of europium on activated charcoal. The percentage adsorption and distribution coefficient (K_D) increase with increasing pH up to a value of 4 and then start to decrease. Maximum adsorption occurs at pH 4 and hence a buffer with a pH of 4 (Fluka, Item No. 82560) was used in all further investigations. The influence of pH on the europium adsorption may be explained as follows. Over the pH range 1–4, competitive adsorption of H_3O^+ and Eu^{3+} on charcoal varies in accordance with the acidity of the solution. As the pH of the solution increases from 1 to 4, the adsorption of H_3O^+ decreases whilst that of Eu^{3+} increases. Over the pH range 5–7, Eu^{3+} ions undergo hydrolysis resulting in the formation of hydroxy ions such as Eu(OH)^{2+}, Eu(OH)^+ and Eu(OH)_3. These hydroxy ions are weakly adsorbed relative to Er^{3+} ions and hence the adsorption of europium on the activated charcoal decreases. Above pH 7, the adsorption process could not be followed because of the formation of insoluble europium complexes (Ichikawa and Sato 1969).

The effect of europium concentration on adsorption has been studied under optimized conditions of 30 min shaking time, pH 4 and 100 mg adsorbent. The concentration of europium was varied from 1000 $\mu$g/ml to 6000 $\mu$g/ml. The results depicted in Figure 4 show that the percentage adsorption and the distribution coefficient (K_D) decreased as the europium concentration increased, indicating that energetically less favourable sites become involved in the process with increasing concentration. The data concerning the dependence of the extent of adsorption on the europium concentration
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were subjected to examination by the Freundlich, Langmuir and Dubinin-Radushkevich (D-R) equations.

![Graph showing % adsorption and K_D vs Eu concentration (µg/ml)]

Figure 4. The effect of europium concentration on its adsorption on to activated charcoal: curve (a), % adsorption; curve (b), K_D.

The Freundlich isotherm equation was used in the form:

\[
\frac{X}{m} = K C_s^{1/n}
\]

where \(X/m\) is the amount of europium ion adsorbed per gram of activated charcoal, \(C_s\) is the equilibrium concentration, \(K\) and \(1/n\) are empirical constants. The Freundlich plot of \(\log(X/m)\) versus \(\log C_s\) shown in Figure 5(a) demonstrates the non-validity of the equation over the whole range of europium concentration.

The Langmuir equation was applied in the form (Afzal et al. 1977):

\[
\frac{C_s}{X/m} = \frac{1}{K_1 K} + \frac{C_s}{K}
\]

where \(X/m\) and \(C_s\) have already been defined, \(K_1\) is a constant related to the heat of adsorption via the relationship \(K_1 = K_0 e^{-q/KT}\), \(q\) is the heat of adsorption and \(K = X_m\) is a measure of the monolayer capacity. A straight line may be obtained by plotting \(C_s/X/m\) versus \(C_s\) [Figure 5(b)], indicated the conformity of the data. Values of the constants \(K\) and \(K_1\) calculated from the slope and intercept of the plot in Figure 5(b) were 25.43 and 9.06, respectively.
The linearized D–R isotherm equation (Aksoyoglu 1989) is:

$$\ln \frac{X}{m} = \ln X_m - K' \varepsilon^2$$

(8)

where

$\varepsilon = RT \ln(1 + 1/C_s)$

$X_m =$ monolayer capacity of adsorbent

$K' =$ constant related to adsorption energy

$R =$ gas constant

$T =$ absolute temperature.

The quantities $X/m$ and $C_s$ have their usual meaning.

A straight line is obtained on plotting $\ln X/m$ versus $\varepsilon^2$ as shown in Figure 5(c), indicating that europium adsorption on to activated charcoal also obeys the D–R equation. Values of $X_m$ and $K'$ calculated from the intercept and slope of the plot were 25.00 and 0.031, respectively. From the value of $K'$ it is possible to calculate the adsorption energy.
E_a using the following equation:

\[ E_a = (-2K')^{-1/2} \]  \hspace{1cm} (9)

when a value of 4.024 kJ/mol was obtained for the adsorption of europium on to activated charcoal.

![Diagram showing the effect of temperature on adsorption of europium on activated charcoal.](image)

Figure 6. The effect of temperature on the adsorption of europium on to activated charcoal.

The dependence of europium adsorption on the temperature was also investigated. The temperature was varied from 10°C to 50°C in 10°C steps while all the other parameters were kept constant. Figure 6 shows that \( K_D \) increases with increasing temperature. This increase may be due to a negative temperature coefficient or to a steep simultaneous decrease in the real adsorption of the solvent (Bikerman 1958). The values of \( \Delta H^0 \) and \( \Delta S^0 \) have been calculated from the slopes and intercepts of the linear variation of \( \ln K_D \) with the reciprocal of the temperature \( 1/T \) (Figure 7) using the relationship:

\[ \ln K_D = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \]  \hspace{1cm} (10)

while \( \Delta G^0 \) values were obtained using the relationship:

\[ \Delta G^0 = \Delta H^0 - T\Delta S^0 \]  \hspace{1cm} (11)

The calculated values of \( \Delta H^0 \), \( \Delta S^0 \) and \( \Delta G^0 \) are listed in Table 1. The values of \( \Delta H^0 \) obtained indicate that europium adsorption on activated charcoal is endothermic.
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![Figure 7. Plots of ln KD versus 1/T for the adsorption of europium on to activated charcoal.](image)

**TABLE 1. Thermodynamic Parameters for the Adsorption of Eu\(^{III}\) on Activated Charcoal**

| Eu conc. (µg/ml) | ΔH\(^{o}\) (kJ/mol) | ΔS\(^{o}\) (kJ/K/mol) | ΔG\(^{o}\) (kJ/mol) |
|-----------------|--------------------|---------------------|--------------------|
| 1000            | 30.23              | 0.162               | 283 K              |
| 2000            | 29.49              | 0.155               | 293 K              |
| 3000            | 20.15              | 0.116               | 303 K              |
| 4000            | 11.89              | 0.082               | 313 K              |
| 5000            | 8.66               | 0.068               | 323 K              |
| 6000            | 7.59               | 0.061               |                    |

One possible explanation for such endothermicity is the well-known fact that the europium ion is well solvated in aqueous media. Hence, in order for the ion to be adsorbed, it must be denuded to some extent of its hydration sheath. This dehydration process requires energy and we assumed that this energy exceeds the exothermicity of ionic attachment to the surface. The assumption which is implied here is that on adsorption the environment of the metal ion is less aqueous than in the solution state. In summary, we may say that the removal of water from the ion is essentially an endothermic process and it appears that the endothermicity of the desolvation process exceeds the
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Figure 8. Variation in the percentage adsorption of europium on to activated charcoal in the presence of different cations.

Figure 9. Variation in the value of $K_D$ for europium adsorption on to activated charcoal in the presence of different cations.
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heat of adsorption by a considerable extent (Qadeer et al. 1993). The values of the free energy change \( \Delta G^0 \) are negative as expected for a spontaneous process. The increase in \(-\Delta G^0\) with increasing temperature shows that europium adsorption on activated charcoal is favoured by higher temperature. This can also be explained on the basis of the above discussion from which it follows that at higher temperatures the ion is more readily desolvated and hence its adsorption become more favourable. Since the adsorption process is endothermic, it follows that under these conditions the process becomes spontaneous because of the positive entropy change.

### TABLE 2. Langmuir and D–R Isotherm Parameters for the Adsorption of Europium on to Activated Charcoal in the Presence of Different Cations and Anions

| Eu in the presence of | Z/r | Langmuir isotherm parameters | D–R isotherm parameters | Cations |
|----------------------|-----|-----------------------------|------------------------|---------|
| Na\(^+\)             | 1.031 | 22.94 8.72                  | 22.83 0.0368            | 3.69    |
| Li\(^+\)             | 1.471 | 21.32 8.30                  | 21.56 0.0490            | 3.19    |
| Zn\(^{2+}\)          | 2.703 | 20.33 6.13                  | 20.31 0.0630            | 2.84    |
| Co\(^{2+}\)          | 2.777 | 19.01 5.21                  | 19.06 0.0792            | 2.51    |
| Ce\(^{3+}\)          | 2.901 | 17.04 4.81                  | 17.13 0.0972            | 2.27    |
| Cr\(^{3+}\)          | 4.762 | 14.51 4.25                  | 14.51 0.1203            | 2.04    |

| Anions              |      |                             |                        |         |
|---------------------|------|-----------------------------|------------------------|---------|
| none                |      |                             |                        |         |
| CH\(_3\)COO\(^-\)    | 25.45| 9.06                        | 25.00 0.0309            | 4.02    |
| S\(_2\)O\(_3\)\(^2-\) | 23.53| 8.34                        | 23.29 0.0375            | 3.65    |
| I\(^-\)              | 22.03| 7.44                        | 21.89 0.0395            | 3.56    |
| Cl\(^-\)             | 20.83| 6.75                        | 20.70 0.0493            | 3.19    |
| NO\(_3\)\(^-\)       | 19.38| 6.28                        | 19.14 0.0543            | 3.03    |
| EDTA                 | 13.25| 1.86                        | 12.38 0.1793            | 1.67    |

The influence of various cations such as Li\(^+\), Na\(^+\), Co\(^{2+}\), Zn\(^{2+}\), Ce\(^{3+}\) and Cr\(^{3+}\) on the adsorption of europium on activated charcoal has also been examined. The concentration of each cation was fixed at 1000 µg/ml and the europium concentration was varied from 1000 µg/ml to 6000 µg/ml. The results of these investigations are shown in Figures 8 and 9. These demonstrate that the greater the ionic potential \((Z/r)\) of the added cation, the smaller the extent to which europium is adsorbed. The Langmuir and D–R isotherm parameters for europium adsorption on to activated charcoal in the presence of different cations have been calculated and are listed in Table 2.
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Figure 10. Variation in the percentage adsorption of europium on to activated charcoal in the presence of various anions.

Figure 11. Variation in the value of $K_D$ for europium adsorption on to activated charcoal in the presence of various anions.
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We have also examined the adsorption behaviour of europium on activated charcoal in the presence of acetate, thiosulphate, iodide, chloride and nitrate anions and in the presence of EDTA. The concentration of each anion was taken as 1000 µg/ml and the concentration of europium was varied from 1000 µg/ml to 6000 µg/ml. The results are shown in Figures 10 and 11. The presence of the acetate ion enhanced the adsorption of europium while the other anions induced a negative effect. The reduction of europium adsorption in the presence of anions was in the order EDTA $> NO_3^- > Cl^- > I^- > S_2O_3^{2-}$. The calculated values of the Langmuir and D-R isotherm parameters for the adsorption of europium in the presence of different anions are listed in Table 2.

**TABLE 3. Percentage Adsorption and $K_D$ Values of Other Metals on Activated Charcoal under the Optimized Conditions for Europium**

| Metal | % Adsorption | $K_D$ (ml/g) |
|-------|--------------|--------------|
| Ce    | 87.92        | 726.45       |
| Eu    | 85.00        | 566.67       |
| Gd    | 81.00        | 426.32       |
| Sm    | 80.16        | 403.78       |
| Er    | 80.15        | 403.78       |
| Dy    | 79.90        | 397.51       |
| U     | 70.46        | 234.45       |
| La    | 56.04        | 127.53       |
| Ga    | 51.93        | 108.12       |
| Y     | 30.02        | 42.86        |
| Cu    | 19.16        | 23.69        |
| Mn    | 15.00        | 17.65        |
| V     | 14.06        | 16.35        |
| Cd    | 14.00        | 16.28        |
| Ni    | 13.82        | 16.01        |
| Ba    | 10.80        | 12.11        |
| Sr    | 10.41        | 11.61        |
| Ru    | 9.16         | 10.07        |
| Cs    | 9.01         | 9.89         |
| Cr    | 8.30         | 9.05         |
| Nd    | 4.40         | 4.60         |
| Pr    | 3.10         | 3.20         |
| In    | 1.00         | 1.01         |

*aConcentration of all metals fixed at 2000 µg/ml.

*bThe order of the entries is on the basis of decreasing adsorption.
To check the selectivity of activated charcoal for europium adsorption, the adsorption of Ce, Er, Gd, Sm, Dy, U, La, Ga, Y, Cu, Mn, V, Cd, Ni, Ba, Sr, Ru, Cs, Cr, Nd, Pr and In on to activated charcoal was examined under the optimum conditions employed for
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europium. The results are listed in Table 3. It is obvious from the data that Ce, Er, Gd, Sm, Dy, U, La, Ga and Y exhibit considerably higher values for the percentage adsorption and $K_D$; hence these would be co-adsorbed alongside europium on to activated charcoal. In contrast, Cu, Mn, V, Cd, Ni, Ba, Sr, Ru, Cs, Nd, Pr and In are poorly adsorbed; hence separation of europium from these metals may be achieved. The separation factor for europium is larger in the presence of Ba, Sr, Ru, Cs, Cr, Nd, Pr and In because these metals have much lower $K_D$ values. The separation of europium in the presence of these metals is illustrated by the bar chart shown in Figure 12.

The feasibility of using activated charcoal for the preconcentration of europium has been further assessed by means of an elution study. This was performed using 3 M HNO$_3$ solution and Figure 13 shows the elution profile of europium obtained. About 96% of the adsorbed europium was recovered by elution with 45 ml of 3 M HNO$_3$ solution.

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