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Retention of Hg(II) by Solid Mercury Sulfide from Acidic Solution

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ABSTRACT

Mercury sulfide was prepared and characterized by measuring average pore size and surface area. The sorption of mercury onto mercury sulfide was investigated in detail with respect to sorptive medium, agitation time, sorbent and sorbate concentration, and temperature. The maximum sorption of mercury ($>$99.8) was achieved from 10^{-3} M hydrochloric acid solution using 50 mg mercury sulfide for 10 minutes. The sorption data of mercury followed the Langmuir isotherm over the entire concentration of mercury investigated whereas the Freundlich and Dubinin–Radushkevich isotherms were obeyed only up to low and moderate concentrations. Useful parameters like sorption capacity and energy have been evaluated using these isotherms. The variation of the equilibrium constant with temperature has the results $\Delta H = 23.8 \pm 3.2$ kJ·mol^{-1}, $\Delta S = 130 \pm 10$ J·mol^{-1}·K^{-1}, and $\Delta G = -13.5 \pm 0.4$ kJ·mol^{-1} at 298 K. Among the ions tested, tartrate, fluoride, and citrate increase the sorption whereas Cu(II), Ba(II), Zn(II), Ni(II), Fe(II), and Cr(III) reduce the sorption significantly. Except for Y(III) ($\sim$91%), all the metal ions showed a lower affinity toward HgS, especially trivalent Ho, Fe, Sc, Tm, Ga, Dy, As, and Pr which showed sorption $>$1 and $<$10%. The mercury sulfide column can be used to separate Hg(II) from these trivalent metal ions.

INTRODUCTION

Mercury and its compounds are used in amalgams, dental preparations, electrical switches and lights, laboratory apparatus and optical equipment,

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metal plating, photography, vapor tubes, and x-ray tubes; and in the manufacture of chlorine, fungicides and germicides, explosives, pigments and paints, catalysts, and pharmaceuticals (1). They are toxic and can enter into body through cuts, by ingestion, or by inhalation. They damage the kidney, the liver, the central nervous system including the brain; the gastrointestinal, genitourinary, and pulmonary tracts; and the eyes and skin. They may cause permanent impairment (2). A large number of inorganic ion-exchangers have been studied to investigate their sorption behavior toward different metal ions (3, 4). In the last decade the sorptive properties of mercury on different inorganic ion-exchangers, including manganese dioxide (5, 6), zirconium oxide (7), and potassium hexatitanate (8), have been investigated. This communication reports the results of a detailed study undertaken to monitor the sorption of Hg onto mercury sulfide, prepared and characterized in this laboratory. This is in continuation of our earlier investigation regarding the sorption of toxic metal ions onto the surface of insoluble sulfides (9). Mercury sulfide has been found to be a useful sorbent for the retention or scavenging of mercury ions from very dilute solutions.

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EXPERIMENTAL

Chemicals and Radiotracers

Mercury sulfide was precipitated by mixing an aqueous solution of mercuric chloride (Merck, Germany) with sodium sulfide (Riedel de Haen, Germany) with occasional stirring. The precipitates were left overnight in the mother liquor and then filtered and washed with deionized water until the washings were neutral (pH $\approx$ 7). The black precipitates were heated in an oven at 105°C for 16 hours. The mercury sulfide thus obtained was used as such as an adsorbent without any further purification. The BET surface area was determined to be $9 \pm 1\ \mathrm{m^2 \cdot g^{-1}}$ by adsorbing nitrogen, and the average pore size was found to be 14.2 μm using a laser micronsizer. The total intrusion volume was measured to be $0.43\ \mathrm{cm^3 \cdot g^{-1}}$ using a mercury porosimeter.

^{203}Hg radiotracer was obtained by irradiating specpure mercuric oxide (Johnson and Matthey) in the PARR-1 research reactor of this Institute at a thermal flux of $4 \times 10^{13}\ \mathrm{n \cdot cm^{-2} \cdot s^{-1}}$ for 5 hours. The oxide was dissolved in dilute hydrochloric acid and stored in 0.1 M HCI. The radiochemical purity was checked by a Canberra multichannel analyzer coupled with a scintillation counter. The specific activity of ^{203}Hg was $2 \times 10^6\ \mathrm{Bq \cdot mL^{-1}}$. The other radionuclides used were also produced by irradiating specpure metals or their appropriate compounds in the same reactor.

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Procedure

The distribution ratio (K_d) and percent sorption have been measured by comparing the initial activity of a ^{203}Hg tracer (CPM/0.5 mL) with the final activity of the solution after equilibrating the mercury solution with mercury sulfide for a specific time interval, normally 10 minutes. The instruments for radiometric assay, pH measurement, shaking, temperature control, and equations used to compute K_d and percent sorption have been given elsewhere (6, 7). The K_d values reported are the average of at least triplicate measurements. The expected errors are ±3%.

RESULTS AND DISCUSSION

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The nature and concentration of an electrolyte affect the sorption behavior of oxides and other surfaces. Therefore, the sorption of mercury onto the surface of its sulfide was first investigated as a function of the concentration of the acids nitric, hydrochloric, perchloric, and sulfuric in the 10^{-1}–10^{-4} M range. The concentration of mercury was kept at 9.29×10^{-5} M. The sorbent, HgS (50 mg), was shaken for 5 minutes with 4 mL of the acid of known concentration containing ^{203}Hg tracer. The results are given in Table 1. Signifi-

TABLE 1
Sorption of Hg(II) (9.29×10^{-5} M) onto HgS (50 mg) after 5 Minutes Shaking from Acidic Solutions

Acid	Concentration (M)	K_d ($cm^3 \cdot g^{-1}$)	% Sorption
HNO_3	10^{-1}	1,979	96.1
	10^{-2}	2,440	96.8
	10^{-3}	16,266	99.5
	10^{-4}	2,048	96.2
HCl	10^{-1}	3,184	97.5
	10^{-2}	3,136	97.5
	10^{-3}	11,963	99.3
	10^{-4}	5,049	98.3
$HClO_4$	10^{-1}	21,747	99.6
	10^{-2}	—	—
	10^{-3}	18,772	99.5
	10^{-4}	26,048	99.7
H_2SO_4	10^{-1}	24,008	99.6
	10^{-2}	6,951	98.8
	10^{-3}	15,846	99.5
	10^{-4}	—	—

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cantly, a 10^{-3} M concentration of all the acids gives a very high distribution ratio (K_d) and percent sorption (>99%). We chose 10^{-3} M hydrochloric acid solution for all subsequent studies. This corresponds to pH 3, where mercury is present as Hg^{2+} (~47%), $HgOH^+$ (~13%), and $Hg(OH)_2$ (~39%). These were estimated by using the hydrolysis constants of mercury (10). These estimates are in agreement with the respective values (43, 13, and 43%) given in the literature (11). Stronger complexes of Hg(II), a soft cation, with chloride ions have been reported (12). Mixed complexes of the type $Hg(OH)_yCl_v^{(2-y-v)+}$ can be expected to form. However, the only species known so far is HgOHCl which has a narrow field of dominance on log[Cl] vs pH diagrams (11). The main species expected in 10^{-3} M HCl would be $HgCl^+$ or $HgCl_2$.

Another parameter which controls the distribution of mercury between sorbent and electrolyte is shaking time. The sorption of mercury onto mercury sulfide has been studied for different intervals of shaking time between 1 and 60 minutes. The concentration of mercury was chosen as 9.92×10^{-5} M and as 50 mg for HgS for this study. The results are graphically depicted in Fig. 1 where sorbed concentration is plotted as a function of shaking time (t) and $\sqrt{t}$. The

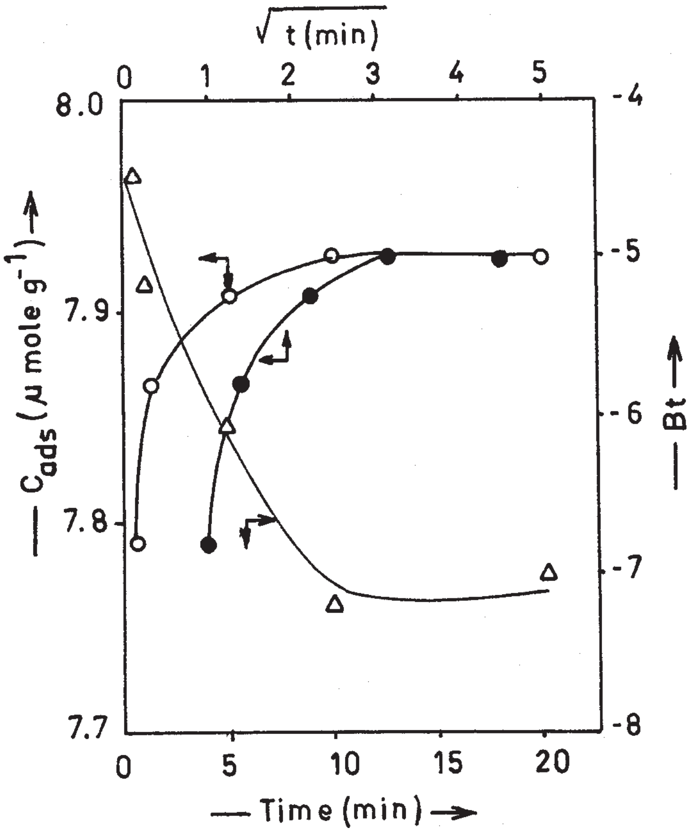

FIG. 1 Sorption of Hg (9.92×10^{-5} M) onto HgS as a function of shaking time (t) and $\sqrt{t}$ from 10^{-3} M HCl solution.

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sorbed concentration starts increasing with an increase in shaking time and attains a constant value within 10 minutes. According to the Morris–Weber equation (13):

$$q_t = R_d \sqrt{t} \tag{1}$$

where q_t is the sorbed concentration at time t, and R_d is the rate constant of intraparticle transport. The plot shown in Fig. 1 is not a straight line, implying that Eq. (1) is not followed by the data and that intraparticle transport does not play any substantial role in the sorption of mercury(II) onto mercury sulfide. In Fig. 1 the values of Bt, which is a mathematical function of $F_t = q_t/q_e$ (q_e = sorbed concentration at equilibrium), can be calculated for each value of F as shown by Reichenburg (14):

$$F = \left(1 - \frac{6}{\pi^2}\right) e^{-Bt} \tag{2}$$

or

$$Bt = -0.4977 - \ln(1 - F)$$

A plot of Bt vs time, also shown in Fig. 1, is not a straight line. This implies that sorption is not entirely controlled by film diffusion.

The effect of the amount of HgS on the sorption of mercury has been studied in the 10–500 mg range from 10^{-3} M HCI solution using 5 minutes of shaking time and a Hg concentration of 1.28×10^{-4} M. Figure 2 gives the results. The distribution ratio (K_d) starts increasing with an increase in the dosage of HgS and attains a maximum value at around 100 mg and then decreases with an increase in the amount of sorbent. A similar trend was observed when the sorption of silver on manganese dioxide (15) was studied as a function of the amount of sorbent for a fixed volume of sorptive solution.

The influence of mercury concentration on its own sorption was also measured over an 8.3-fold range from 6.4×10^{-5} to 5.3×10^{-4} M (Fig. 3). It was found that the distribution ratio is very much affected by the sorbate concentration. These data were then subjected to different sorption isotherms, i.e., Langmuir, Freundlich, and Dubinin–Radushkevich (D–R). The usual equation based on the Langmuir model (16a) represents a homogeneous surface and its low coverage and independent active sites, i.e.,

$$\frac{C_e}{C_{ads}} = \frac{C_e}{Q} + \frac{1}{Qb} \tag{3}$$

was tested. In Fig. 4 the ratio of the equilibrium concentration C_e and the sorbed concentration C_{ads} is plotted against C_e. The Langmuir isotherm is obeyed over the entire concentration range tested. The Langmuir constants Q, related to the sorption capacity, and b, representing the sorption energy, have been computed from the slope and intercept of the plot. The values of Q and

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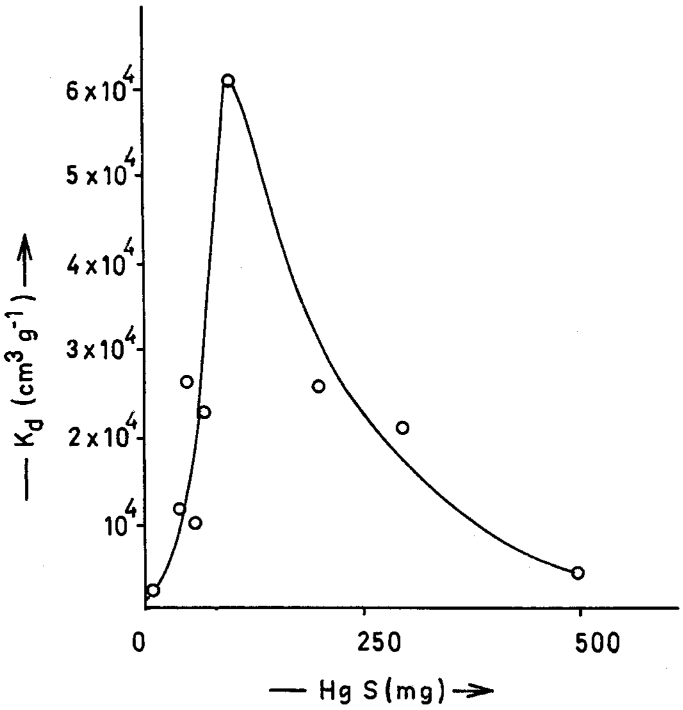

FIG. 2 Variation of distribution ratio (K_d) of Hg (1.28×10^{-4} M) from 10^{-3} M HCl solution with the dosage of sorbent.

of b for the present sorption system are $Q = 0.35 \pm 0.007$ μmol·g^{-1} and of $b = (2.84 \pm 0.27) \times 10^5$ M. This model leads to progressive coverage of the surface by sorbate ions until a monolayer is formed, i.e., until one metal ion is sorbed per active site with the geometric conditions taken into account. The correlation factor for linear regression analysis giving the values of Q and b is 0.99.

The sorption data were also subjected to the Freundlich sorption isotherm (16b), i.e.,

$$C_{\mathrm{ads}} = AC_{\mathrm{e}}^{1/n} \tag{4}$$

where A and $1/n$ are Freundlich parameters signifying sorption capacity and sorption intensity, respectively. This isotherm was tested by plotting log C_{ads} vs log C_{e}. The Freundlich plot is given in Fig. 5. The values of $l/n = 0.39 \pm 0.03$ and of $A = 2.15 \pm 0.09$ mmol·g^{-1} were evaluated for this system.

Another isotherm, i.e., D–R (16c), is expressed as

$$C_{\mathrm{ads}} = X_{\mathrm{m}}e^{-\beta\varepsilon^2} \tag{5}$$

It was tested in the following form:

$$\ln C_{\mathrm{ads}} = \ln X_{\mathrm{m}} - \beta\varepsilon^2 \tag{6}$$

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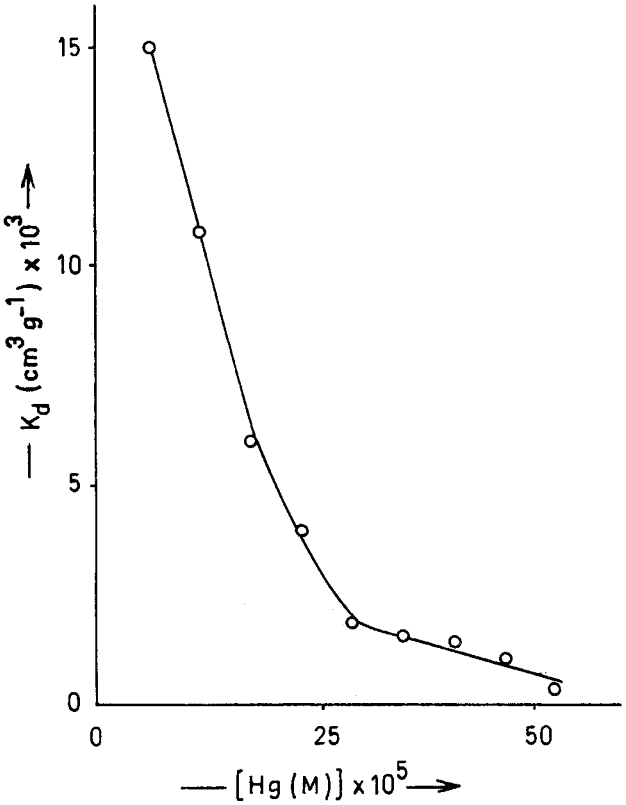

FIG. 3 Sorption of Hg onto HgS (50 mg) as a function of its own concentration from 10^{-3} M HCl solution.

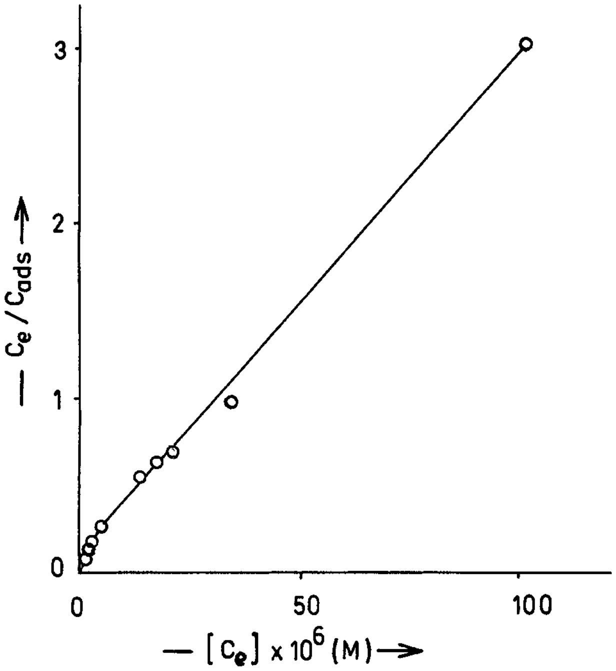

FIG. 4 Langmuir sorption isotherm of Hg onto HgS.

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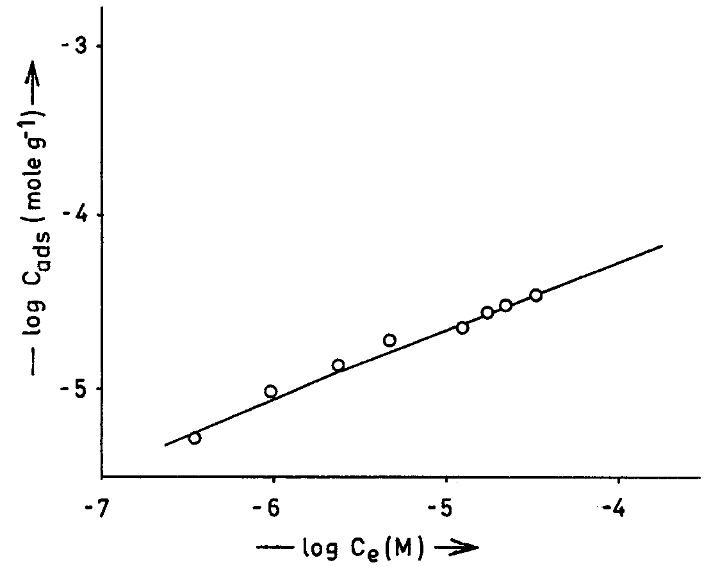

FIG. 5 Freundlich sorption isotherm of Hg onto HgS.

where β is a constant related to energy and ϵ is the Polanyi potential $= RT \ln(1 + 1/C_e)$, where R is a gas constant in kJ·mol^{-1} and T is the temperature in degrees Kelvin. The notations of the other terms used were explained earlier. A plot of 1n C_{ads} vs ϵ^2 is shown in Fig. 6. The values of β and X_m obtained from the slope and intercept are -0.002526 ± 0.000143 mol^2·kJ^{-2} and 0.19 ± 0.03

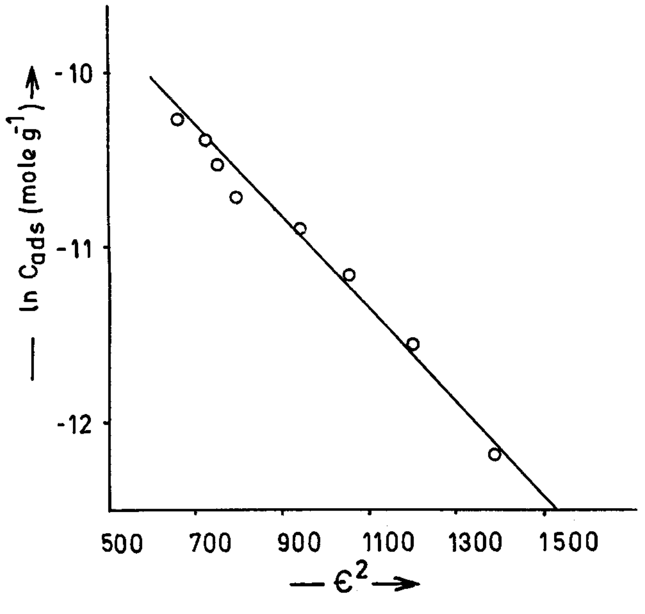

FIG. 6 Dubinin–Radushkevich (D–R) isotherm of Hg onto HgS.

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mmol·g^{-1}, respectively. The value of β can be correlated to energy E when one molecule is transferred from infinity in solution to the surface (17):

$$E = \frac{1}{\sqrt{-2\beta}} \tag{7}$$

The value of E computed from the above equation is 14.2 ± 0.8 kJ·mol^{-1}. It is observed that at higher concentrations of sorbate, both the Freundlich and D–R isotherms are not obeyed. By ignoring C_{ads} values at higher mercury concentrations, the correlation factor (r) for all three isotherms is ≥ 0.99.

The effect of temperature on the sorption of mercury (6.24×10^{-6} M) using 50 mg of the sorbent (HgS) and 5 minutes of shaking time was monitored between 20 and 50°C. The log of the sorption equilibrium constant, $K_c = F_e/(1 - F_e)$, where F_e is the fraction sorbed at equilibrium, was plotted against $1/K$ (Fig. 7). The equilibrium constant (K_c) increases with an increase in temperature. The thermodynamic parameters of ΔH, ΔG, and ΔS were estimated using the following relationships:

$$\log K_c = \frac{-\Delta H}{2.303RT} + \frac{\Delta S}{2.303R} \tag{8}$$

$$\Delta G = -RT \ln K_c \tag{9}$$

The values of $\Delta H = 23.8 \pm 3.2$ kJ·mol^{-1} and $\Delta S = 130 \pm 10$ J·mol·K^{-1} were determined using the slope and intercept of the linear plot shown in Fig. 7. The value of $\Delta G = -13.5 \pm 0.4$ kJ·mol^{-1} at 298 K was computed by employing

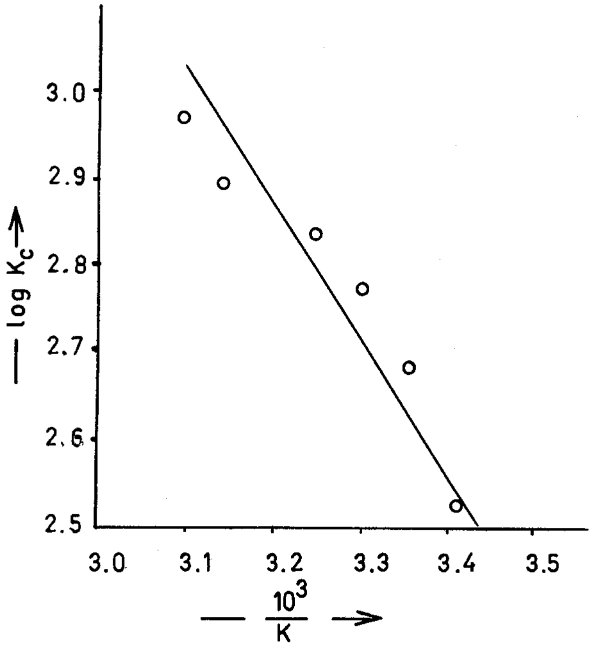

FIG. 7 Variation of sorption equilibrium as a function of temperature.

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Eq. (9). The positive value of ΔH suggests that sorption is endothermic in nature with weak bonding between the sorbent and sorbate, whereas the negative value of ΔG indicates the spontaneity of mercury sorption.

The role of various electrolytes present in a sorptive medium is important in charged surfaces and diffuse layers of counterions in solution. The electrical charge on the sorbent is determined by surface coordination reactions with other specifically sorbing or complex-forming cations and anions. Their competition in equilibrium of sorption, complex formation, dissociation, and precipitation is expected. In order to assess the influence of various common anions and cations, the sorption of mercury was measured in the presence of such additional ions. The anions were added as their sodium or potassium salts whereas the cations were included as their chlorides. Their concentrations were kept at 10 mg/mL whereas the concentration of mercury was $\sim 10^{-4}$ M. The results are listed in Tables 2 and 3. Notably complexing ions like tartrate, fluoride, and citrate enhance the sorption whereas molybdate, cyanide, iodide, thiourea, and thiocyanate cause a reduction in sorption. The enhancement or reduction in sorption in the presence of complexing ions can be interpreted in

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TABLE 2
Influence of Anions on the Sorption of Hg(II) ($\sim 10^{-4}$ M) onto HgS (50 mg) Surface from 10^{-3} M HCl Solution

Additional anion[a]	K_d ($cm^3 \cdot g^{-1}$)	% Sorption
Nil	12,500	99.3
Tartrate	17,174	99.5
Fluoride	16,698	99.5
Citrate	14,898	99.5
Phosphate	8,233	99.0
Ascorbate	8,201	99.0
Oxalate	8,110	99.0
Acetate	7,928	99.0
Sulfate	7,778	99.0
Bromide	1,979	96.1
Thiosulfate	1,413	94.6
EDTA	1,133	93.4
Thiocyanate[b]	595	88.1
Thiourea	563	87.5
Iodide	491	88.1
Cyanide[b]	310	79.4
Molybdate	48.6	37.7

[a] Sodium salt 10 mg/mL.
[b] Potassium salt used.

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TABLE 3
Effect of Cations on the Sorption of Hg(II) ($\sim10^{-4}$ M) onto HgS (50 mg) Surface from 10^{-3} M HCl Solution

Additional cation[a]	K_d ($cm^3 \cdot g^{-1}$)	% Sorption
Nil	12,500	99.3
Ce(III)	3,272	97.6
Zr(IV)	2,996	97.4
Al(III)	2,405	96.8
Co(II)	1,809	95.8
Sr(II)	1,570	95.2
Li(I)	1,233	93.9
Nb(V)	507	86.3
Fe(II)	288	78.0
Tl(I)	148	64.9
Cr(III)	50.7	38.5
Ni(II)	45.0	36.2
Zn(II)	41.0	33.4
Ba(II)	36.6	31.3
Cu(II)	18.7	18.9

[a] 10 mg/mL.

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terms of varying affinity of mercury complexes with such ions toward the sorbent surface. Complexes of mercury with cyanide (10, 12, 18); iodide, thiourea, and thiocyanate (12, 18); and of tartrate (12) and citrate (10, 12) have been reported. All the cations tested tend to reduce the sorption; most significantly are divalent Cu, Ba, Zn, and Ni and trivalent Cr. In the presence of these cations the percent sorption of mercury lies between >18 and <39. These ions ought to be removed from a sorptive solution, otherwise low sorption yields will be obtained. These cations may block the sorption sites or replace mercury ions from the sulfide surface which will no longer be available for the exchange of radioactive mercury ions present in the solution. This is in line with the sorption mechanism envisaged later.

To check the selectivity of the sorbent surface, the sorption of several radionuclides representing different oxidation states and groups has been measured under optimal conditions selected for mercury sorption. The results are given in Table 4 along with their separation factors (α) with respect to Hg(II). All the metal ions studied have a lower sorption than Hg(II). It is obvious that mercury is retained quite selectively over its sulfide surface. Significantly, trivalent Ho, Fe, Sc, Tm, Ga, Dy, As and Pr have shown lower sorption (<10%). In other words, these elements can be separated from Hg(II) by using a bed of mercury sulfide.

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TABLE 4
Sorption of Other Metal Ions onto HgS (50 mg) Surface from 10^{-3} M HCl Solution after Shaking for 5 Minutes

Metal ion	K_d ($cm^3 \cdot g^{-1}$)	% Sorption	$\alpha = K_d(Hg)/K_d(M)$
Hg(II)	12,500	99.4	—
Y(III)	874	91.6	14.30
Sn(II)	44.9	35.4	278.4
Sb(III)	37.1	31.7	336.9
Zn(II)	22.9	22.0	545.8
Sm(III)	19.2	19.4	651.0
Eu(III)	17.4	17.9	718.4
Cr(III)	13.5	14.4	925.9
Co(II)	13.1	14.1	954.2
Er(III)	11.9	12.9	1,050.4
Pr(III)	8.5	9.6	1,468.9
As(III)	7.1	8.2	1,760.6
Dy(III)	6.0	6.9	2,083.3
Ga(III)	4.1	4.9	3,048.8
Tm(III)	3.3	3.9	3,787.9
Sc(III)	2.8	3.2	4,464.3
Fe(III)	2.0	2.5	6,250.0
Ho(III)	1.2	1.7	10,416

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The sorption of Hg(II) may proceed through a metathetical reaction in which a metal sulfide is displaced by an appropriate ion in the solution (19):

$$^{*}Hg^{2+} + HgS \longrightarrow {}^{*}HgS + Hg^{2+}$$

or the sorption of a hydrolyzed species (20):

$$M_{aq}^{m^{+}} + H_2O \overset{\beta_n}{\Leftrightarrow} M(OH)_n^{(m-n)^{+}} + nH^{+}$$

$$S^{-} + M(OH)_n^{m-n^{+}} \overset{\beta_n}{\Leftrightarrow} S^{-}M(OH)_n^{(m-n)^{+}}$$

Mercury sulfide can be effectively used to isolate mercury from industrial effluents and from contaminated water for purification and decontamination. Mercury sulfide can be employed to preconcentrate radionuclides present in large volumes of solutions.

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