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SUBJECT: A Chemical Engineering Development Program - An Investigation of the Kinetic Mechanisms of Uranyl Salt Ion Exchange

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Abstract

Recent literature concerning uranyl salt complex chemistry and ion exchange was reviewed in an effort to develop the present state of understanding of the equilibria and kinetic mechanisms involved. In the light of this, a development program is discussed which hopefully would lead to further enlightenment. Various kinetic mechanisms of sorption and elution are proposed and a comprehensive mathematical development is given for one such sorption mechanism.

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1.0 INTRODUCTION

The purpose of the ion exchange development program is to determine the kinetic mechanisms which are involved in the ion exchange of uranyl salts with anion exchange resins and to formulate design relationships consistent with the facts. These latter relationships are of value in the engineering design and evaluation of fixed bed, fluidized bed, and continuous ion exchange contactors.⁶

The subject program is particularly appropriate at this time in view of recent developments in continuous contacting equipment and the recent advances made in the field of uranyl salt chemistry. Prior to this time the uranyl salt chemistry was thought to be sufficiently complex as to discourage investigations of the sort outlined herein. This conclusion was due in part to Ahrlund's¹ findings that in aqueous solutions the uranyl ion tends to form complexes with anion-polymers which are cationic, neutral, or anionic depending on the complexing strength of the anions. The sulphate and acetate ions, for example, show considerably stronger anionic complexing tendencies than do the bisulphate, nitrate, and chloride ions. The numerous complexes and their equilibria taken together with the usual diffusional mechanisms tend to complicate appreciably the mathematical analysis of the kinetic mechanisms of uranyl ion exchange.

More recent reports by Arden, Wood, Rowley^{2,3} and O'Connor⁸ suggest that at least in the early phases of the present investigations the complications may be lessened by taking account of the effects of pH and concentration on complex formation. At pH two, for example, dilute uranyl solutions containing appreciable sulphate avoid complications that would set in at lower pH values due to excessive concentration of the bisulphate ion⁴; also at pH two one avoids complications that would set in at higher pH values due to hydrolysis of the uranyl ion.

The equilibrium ion exchange studies of Arden et al also suggest that one may adopt the view that at pH 2 or less in a uranyl sulphate system the sulphate and bisulphate ions compete for sites on the ion exchange resin. Simultaneously with this competing process UO_2SO_4 diffuses into the granule

to be sorbed on $\text{SO}_4^{=}$ occupied sites to form $\text{UO}_2(\text{SO}_4)_3^{4-}$. Sorption of UO_2SO_4 on the HSO_4^- occupied sites does not take place because the complexing affinity of UO_2SO_4 for HSO_4^- is negligible. Another way of saying the same thing is that the bisulphate ion resembles the chloride and nitrate ions in its ability to complex with the uranyl group.

The fact that uranyl sulphate will not sorb on HSO_4^- occupied sites thus reducing the equilibrium capacity of the anion exchange resin for uranium suggests as a practical matter in economics that uranyl sulphate anion exchange investigations at pH less than two might not be worth while for other than academic reasons.

Another important conclusion from the foregoing observations is that UO_2SO_4 sorption from a bisulphate free solution on sulphate sites may be a unilateral process. The bonding in the UO_2SO_4 sorption is sufficiently strong and the chemical reaction involved in the sorption process is sufficiently complete that uranium may not be appreciably desorbed in sulphate solution (of low HSO_4^- concentration) for example by simply reducing the uranyl concentration in solution. Because of the unilateral process a saturation wave front moves into the granule during sorption. Behind this wave front the $\text{SO}_4^{=}$ sites are saturated with UO_2SO_4 while in front of it there is no UO_2SO_4 either sorbed or dissolved in the water of hydration of the granule. In the region of resin matrix saturation, i.e., the region of saturated sulphate sites there exists a concentration gradient of UO_2SO_4 dissolved in the water of hydration of the resin particle. It is this gradient that supports diffusion of the UO_2SO_4 and consequently the migration of the saturation wave front to the innermost regions of the anion exchange resin.

A desorption process which is to be successful in the removal of uranium from an anion exchange resin must involve an adequate constituent such as NO_3^- or Cl^- which will compete with the original sulphate ions for sites on the resin particle. By so doing, the basic structure of the $\text{UO}_2(\text{SO}_4)_3^{4-}$ on the resin will be undermined and both the $\text{SO}_4^{=}$ ions and UO_2SO_4 will be liberated to diffuse out of the resin particle and to be carried away by the solution.

The ease of desorption will depend upon the NO_3^- or Cl^- ion concentration in solution. The more concentrated solutions are the more favorable because the separation factor for the monovalent-divalent competition is governed by the quantity:

$$K_1 \frac{C_v}{C_o}$$

where

C_v = the equivalent capacity of the resin

C_o = the total equivalent anion concentration in solution

K_1 = a constant

This quantity suggests, and experiments confirm, that ion exchange resin is much more selective for polyvalent ions over monovalent ions in dilute solution than in concentrated ones.

From the standpoint of an organized development program, it is probably better to investigate the sorption mechanism first and the desorption last. In the sorption investigations the work at least in the early stages should be limited to pH two, dilute uranyl sulphate concentrations, and fairly high sulphate concentrations to suppress the HSO_4^- ion. The incremental fixed bed and perhaps longer ones may be used to investigate the effects on the expiration curves of particle size, fluid flow rate, uranyl concentration, cross linkage, temperature. A fluidized single stage may also be used to good advantage.

The purpose of the investigations may be more specifically defined as being that of developing either analytically or by machine computer techniques the solutions to the kinetic problems and then to look for coincidence between the results predicted by these and the actual experimental results. Coincidence would tend to confirm the postulated kinetic mechanism and the associated solution, the latter being unimportant for its utility as the design relationship governing the process. It is understood, of course, that the foregoing investigation must be accompanied by appropriate equilibrium studies.

2.0 PRESENT STATUS OF THE MATHEMATICAL ANALYSIS

Thus far the analysis of kinetic mechanisms has been confined to the uranyl sulphate sorption process at pH = 2 as a practical matter. The simplest model envisions resin, initially in the SO_4^- form, being bathed in a solution of UO_2^{++} , UO_2SO_4 , $\text{UO}_2(\text{SO}_4)_2^{--}$, $\text{UO}_2(\text{SO}_4)_3^{--4}$ with only the UO_2SO_4 molecules diffusing into the resin in appreciable quantity.

The resin is assumed to be a gel consisting of an organic matrix with water of hydration distributed throughout the matrix. The resin surface concentration $C_s^*(t)$, of the UO_2SO_4 in the water of hydration is given by the expression

$$C_s^*(t) = \frac{\alpha}{1 - \alpha} C_c^* \quad (1)$$

where

α = void fraction or ratio of interstitial fluid between resin granules to the volume of the dense bed.

$C_{c-\text{UO}_2\text{SO}_4}^*$ = the uranyl sulphate concentration in solution at the solution-resin granule interface.

The concentration of UO_2SO_4 in the bulk solution flowing through a fixed dense bed is $C_{c-\text{UO}_2\text{SO}_4}$. The concentration of the UO_2SO_4 in the water of hydration at any point in a granule is $C_s(r, t)$.

For diffusion into the granule to take place:

$$C_s^* > C_s$$

Also if the equivalent capacity of the resin matrix is C_R then the matrix molar $\text{UO}_2(\text{SO}_4)_3^{--4}$ concentration is:

$$\frac{C_R}{4} \quad R \geq r \geq a$$

$$0 \quad a > r \geq 0$$

where:

R = radius of the granule assumed to be spherical

r = radial position in the granule

$a = a(t)$ is the radial position of the matrix saturation wave front at any time t .

Also:

$$C_s = 0 \quad r = a$$

The diffusion equation for the spherical granule is

$$\frac{\partial U}{\partial t} = D \frac{\partial^2 U}{\partial r^2} \quad (2)$$

where:

$$U(r, t) = r C_s(r, t) \quad (3)$$

D = the diffusivity UO_2SO_4 through the granule

Equation (2) is subject to the limiting conditions that:

$$\begin{aligned} C_s &= C_s^* & r &= R \\ C_s &= 0 & r &= a \\ C_s &= 0 & t &= 0 \end{aligned}$$

and the saturation wave front moves according to the relation:

$$\frac{4D}{C_R} \left[\frac{\partial C_s}{\partial r} \right]_{r=a} = - \frac{da}{dt} \quad (4)$$

If the rate of accumulation of UO_2SO_4 in the water of hydration is small (as is the case) then equation (2) reduces to:

$$\frac{\partial^2 U}{\partial r^2} = 0 \quad (5)$$

which subject to the boundary conditions has the solution:

$$C_s = C_s^* \frac{\frac{1}{a} - \frac{1}{r}}{\frac{1}{a} - \frac{1}{R}} \quad (6)$$

Substituting equation (6) in (4) one finds that:

$$\frac{4D}{C_R} \frac{C_s^*(t)}{\left[a - \frac{a^2}{R} \right]} = - \frac{da}{dt} \quad (7)$$

Equation (5) has the effect of expediting an otherwise insuperable mathematical situation which has been discussed quite thoroughly by Lightfoot⁷, Carslaw and Jaeger⁵ and others. Actually equation (2) with the limiting conditions and equation (4) constitutes a non-linear problem of a sort that is even difficult to cope with on machine computers^{9,10} when account is taken of the fact that the bed material balances and chemical equations must be solved simultaneously with it. By integration of equation (7)

$$\frac{R^2}{6} \left[1 + 2 \left(\frac{a}{R} \right)^3 - 3 \left(\frac{a}{R} \right)^2 \right] = \frac{4D}{C_R} \int_0^t C_s^*(t) dt \quad (8)$$

Equation (8) is important in the present analysis because it defines "a" as a function of "t" which hitherto had not been available in explicit form. Equation (6) is important because it also permits the evaluation of the rate equation:

$$\frac{\bar{R}}{1 - \alpha} = \frac{\partial Q}{\partial t} = \frac{3D}{R} \left[\frac{\partial C_s}{\partial r} \right]_{r=R} \quad (9)$$

for diffusion of UO_2SO_4 through the granule, thus:

$$\frac{\bar{R}}{1 - \alpha} = \frac{3D}{R^3} \frac{C_s^*(t)}{\frac{1}{a} - \frac{1}{R}} \quad (10)$$

where:

$$Q = \frac{\text{total moles } UO_2SO_4 \text{ in granule}}{ft^3 \text{ of granule}}$$

The rate equation for diffusion through the laminar liquid film surrounding the granule is:

$$\frac{\bar{R}}{\alpha} = (k_L a) \left[C_{c-UO_2SO_4} - C_{c-UO_2SO_4}^* \right] \quad (11)$$

where at the fluid solid interface:

$$C_s^*(t) = \frac{\alpha}{1 - \alpha} C_{c-UO_2SO_4}^* \quad (12)$$

and

$(k_L a)$ = the fluid phase transfer coefficient, time⁻¹ (Note that "a" used here is not the radial position of the wave front).

Eliminating the star quantities from equations (10-12) one finds that the over all rate equation is:

$$\frac{\bar{R}}{\alpha} = \frac{C_{c-UO_2SO_4}}{\frac{R^3}{3D} \left[\frac{1}{a} - \frac{1}{R} \right] + \frac{1}{\alpha(k_L a)}} \quad (13)$$

Also from equations (8), (11), and (12):

$$1 + 2\left(\frac{a}{R}\right)^3 - 3\left(\frac{a}{R}\right)^2 = \frac{24\alpha \cdot D}{(\alpha - 1)C_R R^2} \int_0^t \left[C_{c-UO_2SO_4} - \frac{\bar{R}}{\alpha(k_L a)} \right] dt \quad (14)$$

Equation (13) and (14) must be solved simultaneously with the material balances for the bed the latter being derived based on the diagram of an elemental section of bed dZ shown in Fig. 1.

Figure 1

Now as dy moles of UO_2SO_4 are removed from the fluid as it passes from Z to $Z + dZ$ the solution equilibrium is upset according to the equation:

where the equilibrium constant is:

$$K = \frac{C_{\text{c-UO}_2\text{SO}_4}^2}{C_{\text{c-UO}_2} C_{\text{c-UO}_2(\text{SO}_4)_2}} \quad (16)$$

To re-establish equilibrium:

$2 dx$ moles UO_2SO_4 formed

dx moles $\text{UO}_2(\text{SO}_4)_2$ reacted

dx moles UO_2 reacted

Thus:

$$dC_{\text{c-UO}_2} = -dx \quad (17)$$

$$dC_{\text{c-UO}_2(\text{SO}_4)_2} = -dx \quad (18)$$

$$dC_{\text{c-UO}_2\text{SO}_4} = 2 dx - dy \quad (19)$$

where the signs are determined from Fig. 2.

Figure 2

Using the Euler expansion for the total derivatives of concentration in equations (17-19) these may be expanded thus:

$$\frac{\partial c_{c-UO_2}}{\partial \theta} = -v_c \frac{\partial c_{c-UO_2}}{\partial Z} - \frac{dx}{d\theta} \quad (20)$$

$$\frac{\partial c_{c-UO_2(SO_4)_2}}{\partial \theta} = v_c \frac{\partial c_{c-UO_2(SO_4)_2}}{\partial Z} - \frac{dx}{d\theta} \quad (21)$$

$$\frac{\partial c_{c-UO_2SO_4}}{\partial \theta} = -v_c \frac{\partial c_{c-UO_2SO_4}}{\partial Z} + 2 \frac{dx}{d\theta} - \frac{dy}{d\theta} \quad (22)$$

where

$$v_c = \frac{dZ}{d\theta} = \frac{Q_c}{\alpha A} = \text{interstitial fluid velocity} \quad (23)$$

$$t = \theta - \frac{Z}{v_c} \quad (24)$$

and

$$\frac{dy}{d\theta} = \frac{\bar{R}}{\alpha} \quad (25)$$

Since the individual granule contact time, t , is related to the total run time, θ , by equation (24), one obtains, according to the Euler theorem:

$$\left(\frac{\partial c}{\partial \theta}\right)_Z = \left(\frac{\partial c}{\partial t}\right)_Z \left(\frac{\partial t}{\partial \theta}\right)_Z + \left(\frac{\partial c}{\partial Z}\right)_t \left(\frac{\partial Z}{\partial \theta}\right)_Z = \left(\frac{\partial c}{\partial t}\right)_Z \quad (26)$$

and

$$\left(\frac{\partial c}{\partial Z}\right)_\theta = \left(\frac{\partial c}{\partial t}\right)_Z \left(\frac{\partial t}{\partial Z}\right)_\theta + \left(\frac{\partial c}{\partial Z}\right)_t \left(\frac{\partial Z}{\partial Z}\right)_\theta = -\frac{1}{v_c} \left(\frac{\partial c}{\partial t}\right)_Z + \left(\frac{\partial c}{\partial Z}\right)_t \quad (27)$$

thus:

$$\left(\frac{\partial c}{\partial \theta}\right)_t + v_c \left(\frac{\partial c}{\partial Z}\right)_\theta = v_c \left(\frac{\partial c}{\partial Z}\right)_t \quad (28)$$

In view of this general relationship between the two coordinate systems equations (20-22) may be written in the form:

$$v_c \frac{\partial C_{c-UO_2}}{\partial Z} = - \frac{dx}{d\theta} \quad (29)$$

$$v_c \frac{\partial C_{c-UO_2(SO_4)_2}}{\partial Z} = - \frac{dx}{d\theta} \quad (30)$$

$$v_c \frac{\partial C_{c-UO_2SO_4}}{\partial Z} = 2 \frac{dx}{d\theta} - \frac{\bar{R}}{\alpha} \quad (31)$$

From equations (29) and (30) it follows that:

$$C_{c-UO_2} - i - C_{c-UO_2} = C_{c-UO_2(SO_4)_2} - i - C_{c-UO_2(SO_4)_2} \quad (32)$$

where i refers to initial concentrations. Also from stoichiometry one knows that:

$$C_c = C_{c-UO_2} + C_{c-UO_2SO_4} + C_{c-UO_2(SO_4)_2} \quad (33)$$

where:

C_c = total molar U concentration in solution at any point in the column.

The derivative of the natural log of equation (16) is

$$\frac{dC_{c-UO_2(SO_4)_2}}{C_{c-UO_2(SO_4)_2}} + \frac{dC_{c-UO_2}}{C_{c-UO_2}} = 2 \frac{dC_{c-UO_2SO_4}}{C_{c-UO_2SO_4}} \quad (34)$$

Combining equations (16), (17), (18), (19), and (34) one obtains:

$$\left[\frac{C_{c-UO_2SO_4}}{C_{c-UO_2}} + K \frac{C_{c-UO_2}}{C_{c-UO_2SO_4}} + 2 \right] \frac{dx}{d\theta} = \frac{\bar{R}}{\alpha} \quad (35)$$

Equations (29) and (31) may be combined to show that:

$$\frac{\partial [C_{c-UO_2SO_4} + 2C_{c-UO_2}]}{\partial Z} = - \frac{\bar{R}}{\alpha v_c} \quad (36)$$

Equations (29) and (35) may also be combined to show that:

$$\left[\frac{C_{c-UO_2SO_4}}{C_{c-UO_2}} + K \frac{C_{c-UO_2}}{C_{c-UO_2SO_4}} + 2 \right] \frac{\partial C_{c-UO_2}}{\partial Z} = - \frac{\bar{R}}{\alpha v_c} \quad (37)$$

Thus the ultimate solution to the original problem has been reduced to a simultaneous solution of equations (13), (14), (36), and (37), subject to the fixed bed limiting conditions:

$$a = R \qquad t = 0$$

$$C_{c-UO_2} = C_{c-UO_2} - i \qquad Z = 0$$

$$C_{c-UO_2(SO_4)_2} = C_{c-UO_2(SO_4)_2} - i \qquad Z = 0$$

$$C_{c-UO_2SO_4} = C_{c-UO_2SO_4} - i \qquad Z = 0$$

As a matter of convenience it may be noted that equations (36) and (37) may be combined to yield:

$$\frac{\partial C_{c-UO_2SO_4}}{\partial C_{c-UO_2}} = \frac{C_{c-UO_2SO_4}}{C_{c-UO_2}} + K \frac{C_{c-UO_2}}{C_{c-UO_2SO_4}} \quad (38)$$

If this equation is multiplied by:

$$\frac{C_{c-UO_2SO_4}}{C_{c-UO_2}}$$

it may be integrated as a linear first order equation to show that:

$$\left[\frac{C_{c-UO_2SO_4}}{C_{c-UO_2}} \right]^2 - \left[\frac{C_{c-UO_2SO_4} - i}{C_{c-UO_2} - i} \right]^2 = 2K \ln \frac{C_{c-UO_2}}{C_{c-UO_2} - i} \quad (39)$$

From the solution to the problem one obtains C_{c-UO_2} or $C_{c-UO_2SO_4}$ or both as a function of Z and t . This information with equations (32) and (39) would permit calculating C_c from equation (33). It is C_c , the total molar uranium concentration, that is actually measured experimentally.

At the present time it appears that the solution to the problem will have to be obtained through machine computation. In the event an analog computer is used the bed could be thought of as being divided into N segments each ΔZ long.

Equation (36) could be written:

$$\frac{\Delta C_{c-UO_2SO_4}}{\Delta Z} + 2 \frac{\Delta C_{c-UO_2}}{\Delta Z} = \frac{\bar{R}}{\alpha v_c} \quad (40)$$

where:

$$\Delta Z = \frac{L}{N}$$

L = total fixed bed length

and in general:

$$\Delta C = C_{n+1} - C_n$$

where:

n = the number of the segment under consideration

If this procedure is followed, then one might want to feed equation (39) into a function generator and omit equation (37) from further consideration. Otherwise, equation (37) would also have to be converted to finite difference form and inserted into the computer. In any event "t" is the natural time base of the computer.

3.0 OTHER KINETIC MECHANISMS

Although under the circumstances the foregoing kinetic mechanisms for sorption and desorption seem most plausible, they are not the only ones possible. Some other possibilities are listed as follows:

- (a) A variation on the foregoing mechanisms might involve demanding an exact solution to equation (2) and then using this solution in place of equation (6).
- (b) Another variation would be to assume a reversible process as a substitute for the unilateral process discussed in the text.
- (c) Another variation would be to assume that $UO_2(SO_4)_2$ and $UO_2(SO_4)_3^{4-}$ also diffuse freely in the granule and enter into the unilateral process.

- (d) Another variation might be based on the desire to work in the pH range less than two. The simultaneous competition for resin sites between $\text{SO}_4^{=}$ and $\text{HSO}_4^{=}$ would have to be taken into account.
- (e) The most drastic change in kinetic mechanism would result if one discarded the unilateral sorption process and assumed true ion exchange instead wherein the various ions were assumed to behave as simple ions. This would involve considerable complication because of the numerous possibilities for simultaneous exchange and shift in equilibrium among ions. Somehow the end result would have to amount to the same thing as for the unilateral process in order to be consistent with what appears to be experimental fact.

The desorption kinetic mechanism involving $\text{NO}_3^{=}$, Cl^{-} ion eluant has already been discussed in the text. A rather simple competition between say $\text{SO}_4^{=}$ and $\text{NO}_3^{=}$ for resin sites is anticipated. UO_2SO_4 would remain at only those $\text{SO}_4^{=}$ occupied sites that had not been replaced by $\text{NO}_3^{=}$. In solution one might assume that any complexing of UO_2^{++} that takes place does so only with the $\text{SO}_4^{=}$ and not the $\text{NO}_3^{=}$ because of the overwhelming complexing strength of the $\text{SO}_4^{=}$ ion.

4.0 LIST OF SYMBOLS

$C_g(r,t)$ = concentration of UO_2SO_4 in the water of hydration of a resin particle, moles/ft³ of particle.

r = radial position in a granule of resin, ft.

t = time a given particle is in contact with uranyl sulphate solutions, hr.

$U = r C_g(r,t)$.

R = radius of granule, ft.

$a(t)$ = the radial position of the matrix saturation wave front which passes into granule or proceeds toward the center of the granule with time, ft.

D = diffusivity of UO_2SO_4 through granule, sq ft/hr.

C_R = equivalent capacity of resin, equivalents/cu ft of resin particle.

C_c = total molar uranium concentration in solution or continuous phase, moles per cu ft solution.

C_{c-UO_2} = molar concentration of U in UO_2^{2+} form in solution, moles/cu ft.

$C_{c-UO_2SO_4}$ = molar concentration of U in UO_2SO_4 form in solution, moles/cu ft.

$C_{c-UO_2(SO_4)_2}$ = molar concentration of U in $UO_2(SO_4)_2^{2-}$ form in solution, moles/cu ft.

Z = distance along column measured from feed end, ft.

Q = total moles UO_2SO_4 in a granule per cu ft granule (an average molar matrix and water of hydration concentration).

α = cu ft of continuous phase solution per cu ft of bed.

$$\bar{R} = (1 - \alpha) \frac{\partial Q}{\partial t}$$

A = cross area of tower, sq ft.

θ = total time a flow experiment has run, hr. θ differs from t in that

$$t = \theta - \frac{Z}{v_c}$$

v_c = interstitial velocity of continuous phase.

$$Q_c = \alpha A v_c$$

K = equilibrium constant. If a subscript on K is used, it is to denote a particular reaction as shown in text.

C_{c-i} = an initial concentration in solution of an ion indicated by an additional subscript on C_{c-i} for example C_{c-UO_2-i}

$$dy = \frac{\bar{R}}{Q} d\theta$$

$$dx = -dC_{cUO_2} = -dC_{cUO(SO_4)_2}$$

Asterisk quantities refer to resin - solution interface.

$(k_L a)$ = liquid film transfer coefficient, $\frac{1}{hr}$ (note that "a" in bracket is not a $[t]$).

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