Recovery of Uranium from Seawater Using Amidoxime Hollow Fibers

A novel amidoxime-group-containing adsorbent of hollow-fiber form (AO-H fiber) was prepared by radiation-induced graft polymerization of acrylonitrile onto a polyethylene hollow fiber, followed by chemical conversion of the produced cyano group to an amidoxime group. Distribution of the amidoxime group was uniform throughout the hollow-fiber membrane. The fixed-bed adsorption column, 30 cm in length and charged with the bundle of AO-H fibers, was found to adsorb uranium from natural seawater at a sufficiently high rate: 0.66 mg uranium per g of adsorbent in 25 days.

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Introduction

The concentration of uranium in seawater is remarkably constant at 3.3 mg U/m^3. The main uranium species in seawater is the stable anionic tricarbonate-uranyl complex, $UO_2(CO_3)_3^{4-}$ (Saito and Miyauchi, 1981, 1982). Marine uranium displays no detectable deviation from the normal terrestrial U-235/U-238 isotope ratio. The total volume of the oceans has been estimated to be 1.37×10^{18} m^3, and the dissolved uranium should amount to 4.5×10^9 tons, which is almost a thousandfold larger than the terrestrial resources of reasonable concentrations. Thus, the ocean is virtually a limitless reservoir of dissolved uranium in a well-defined chemical environment.

A recovery program was begun in England in the early 1960's (Davies et al., 1964). Later some studies were made by universities and by private companies in various countries, including Japan and West Germany (Schenk et al., 1982; Astheimer et al., 1983). Many methods of recovery have been suggested: coprecipitation, adsorption, ion flotation, solvent extraction, and others. Of these, only the adsorption process using a suitable adsorbent seemed to be feasible (Kanno, 1981).

Considerable effort has been directed toward developing such adsorbents that originate from inorganic or organic materials. Hydrous titanium oxide was identified as the most promising inorganic adsorbent. Organic adsorbents were classified into three types: a chelating ion-exchange resin containing amidoxime functional groups (Egawa et al., 1980a,b), a macrocyclic compound bounded to a resin (Tabushi and Kobuke, 1981), and a cellulose resin immobilized by polyphenol compounds (Sakaguchi and Nakajima, 1987). Loading capacities or adsorption isotherms reported so far in natural seawater are summarized in Figure 1 and Table 1. Of the organic adsorbents, intensive studies have been made on the amidoxime-group-containing, chelating resin because of its ease of synthesis. Various shapes of amidoxime resin, such as spheres (Sakane et al., 1982; Takagi et al., 1986), membranes (Saito et al., 1987a,b), and fibers (Katoh et al., 1982a,b), have been prepared.

Since the concentration of uranium in seawater is extremely low, a large volume of seawater must be contacted with the adsorbent to recover significant quantities of uranium. The contacting mode can be divided into three types according to the force transporting the seawater: pumping, direct utilization of ocean current, or wave utilization. Moreover, the adsorption bed is classified as a fixed bed or a fluidized bed. Adsorption bed performance already reported is summarized in Table 2. In this table, the adsorbent synthesized by GIRIS exhibits the highest adsorption rate. This is due to the extremely hydrophilic properties of amidoxime resins. However, it does not satisfy the requirements for mechanical strength and chemical durability to be applied in practice.

The advantages of the radiation-induced grafting method are as follows:

1. It is applicable to any shape of the base polymer
2. Grafted polymer chains on chemically and mechanically stable base polymers can be functionalized
3. Any catalysts in the polymerization process are unnecessary

Since a microporus hollow fiber provides a high surface-area to volume ratio and high porosity for grafting, it is expected to be a favorable base polymer for preparing the adsorbent for the

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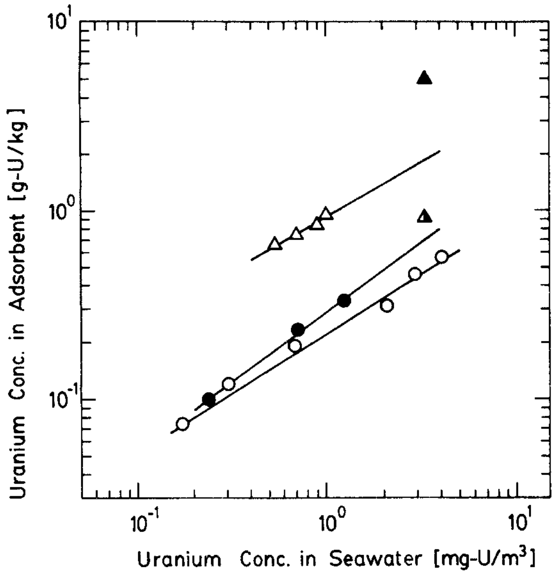

Figure 1. Summary of adsorption isotherms reported.
Symbols key in Table 1.

recovery of uranium from seawater. In addition, the fixed bed charged with hollow fibers has a significant advantage in that it has a pressure drop about an order lower than the corresponding pressure drop in a bed charged with spherical adsorbents, therefore it is suitable for contact with a tremendous volume of seawater.

The objectives of our study were twofold:

1. To prepare the amidoxime-group-containing adsorbent with a hollow-fiber form by radiation-induced grafting.
2. To propose a novel fixed bed charged with the hollow fibers and examine the performance of the adsorption bed with a low flow resistance by measuring the adsorption rate and pressure drop.

Theory

Mass balance in the fixed bed can be expressed by the following equation:

$$-\partial U/\partial x = \partial Q/\partial \tau \qquad (1)$$

Adsorption rate may be represented according to the linear driving force (LDF) model:

$$\partial Q/\partial \tau = \kappa(U - U^*) \qquad (2)$$

where U and U^* are respectively the concentration in solution and that in equilibrium with Q, in dimensionless form. The adsorption isotherm can be written by the Freundlich-type equation:

$$Q = U^{*1/n} \qquad (3)$$

The dimensionless parameter given in Eq. 2 is:

$$\kappa = K_F a_v L/u \qquad (4)$$

The initial and boundary conditions are:

$$\text{at } \tau = 0 \qquad Q = 0 \qquad (5)$$

$$\text{at } x = 0 \qquad U = 1 \qquad (6)$$

These equations can be solved numerically by using the finite-difference method (von Rosenberg et al., 1977; Suzuki, 1983). The dimensionless time and height increments are taken as 0.02 and 0.05, respectively. The results of adsorption rate for the case of the Freundlich constant, $n = 5$, are shown in Figure 2.

To examine the order of $K_F a_v$, an extreme case where the liquid-film mass transfer is controlling was considered. The correlation equation for the liquid-film mass transfer coefficient inside the hollow fiber, k_f, is given by Yang and Cussler (1986):

$$k_f d_i/D = 1.64\,(u d_i^2/\epsilon L D)^{0.33} \qquad (7)$$

Substituting physical properties and operating conditions of this study into this equation, κ was calculated between 4.8 and 12. A dimensionless contact time, $\tau = 1$, was calculated to be required until an adsorption equilibrium was attained. Since the concentration factor, q_0/C_0, of the amidoxime resin in seawater is predicted to be more than 10^6, which is one or two orders larger than conventional adsorbents such as activated carbon, several hundreds of days are required to determine the adsorption isotherm. Therefore, difficulty in the kinetic analysis of the adsorption rate data is characteristic of research on the recovery of uranium from seawater.

Experimental Procedure

Preparation of adsorbent

Porous polyethylene hollow fiber (Mitsubishi Rayon Co., Japan) was used as the base polymer for grafting. The inner and outer diameter of this hollow fiber are 0.027 and 0.038 cm,

Table 1. Summary of Adsorption Isotherms Reported

Key for Fig. 1	Isotherm	Temp. K	Adsorbent*	Ref.
○	$q/0.47 = (C/3.3)^{1/1.6}$	305	T	Ogata (1980)
●	$q/0.70 = (C/3.3)^{1/1.35}$	298	T	Kanno (1983)
▲	$q = 5$ at $C = 3.3$	—	O	Tabushi and Kobuke (1981)
△	$q/1.86 = (C/3.3)^{1/1.72}$	303	O	Sakaguchi and Nakajima (1987)
▲	$q = 0.93$ at $C = 3.3$	—	O	Chen (1981)

*T, Inorganic adsorbent containing hydrous titania
O, Organic resin

Table 2. Previous Studies on Adsorption Rate

Ref.	Bed*	*dc* cm	*Lh* cm	*u* cm/s	Adsorbent**	Shape† mm	Adsorption Rate g U/kg/10 days
Astheimer et al. (1983)	CFL	4 7.35	44	0.29–1.36	AO	Sp 0.2–1.0	0.19 (301 K)
Wen and Huang (1983)	F	3.3	<15	0.30	T	Sp 0.8–1.1	0.073 (295 K)
Omichi et al. (1986b)	F	10	<50	0.044–0.44	AO	Fiber	0.14
Kanno (1983)	FL			0.5	T	Sp 0.3–1.0	
Egawa (1986)	FL	6.5			AO	Sp 0.25–0.5	0.14
GIRIS (1980)	F	2.5	0.3	0.34	AO	Fiber	1.8 (298 K)
Uezu et al. (1987)	F	3.1	30	0.125–1	AO	CF	0.11 (298 K)
This study	F	3.1	30	0.25–1	AO	HF	0.26 (298 K)

*F, fixed bed; FL, fluidized bed; CFL, conical fluidized bed
**T, inorganic adsorbent containing hydrous titanium
AO, organic adsorbent containing amidoxime group
†Sp, spherical; HF, hollow fiber; CF, capillary fiber

respectively. The preparation process of the chelating resin adsorbent by radiation-induced graft polymerization is detailed by Okamoto et al. (1985) and Omichi et al. (1985, 1986a,b). The base polymer was irradiated by an electron beam at ambient temperature in a nitrogen atmosphere. Immediately after irradiation, the bundle of hollow fibers was reacted in a vapor of deaerated acrylonitrile. Vaporized acrylonitrile was reacted with the trapped radicals in the base polymer. The cyano group of the grafted chains was converted into the amidoxime group by reaction with hydroxylamine. Detailed reaction conditions of both grafting and amidoximation are listed in Table 3. A more detailed description of amidoximation has been previously published (Hori et al., 1986, 1987). The amidoxime-group-containing, polyethylene-based hollow fiber will be hereafter referred to as the AO-H fiber.

The amount of the amidoxime group was calculated from the amount of HCl adsorbed. To examine the distribution of the introduced functional groups across the AO-H fiber, samples of the AO-H fibers were soaked in cupric chloride solution. After equilibration, an X-ray microanalysis (XMA) of the AO-H fiber was used to determine the extent of copper adsorption.

Table 3. Reaction Conditions of Grafting and Amidoximation

Grafting	
Irradiation dose	20 Mrad
Reaction temp.	308 K
Amidoximation	
Conc. $NH_2OH \cdot HCl$	3%
Reaction temp.	323 K
Reaction time	10 h

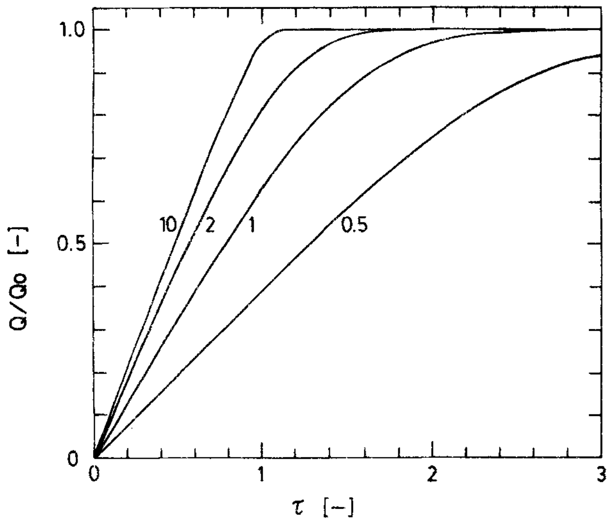

Figure 2. Dimensionless amount of uranium adsorbed vs. dimensionless time.

Values *K* defined in Eq. 4.

Prior to the adsorption experiments, AO-H fibers were soaked in 2.5% KOH solution at 323 K for 1 h and then rinsed repeatedly with deionized water until the pH of the washings was about 9. The dimensions of AO-H fiber conditioned with KOH solution were measured with a microscope.

Adsorption rate

A diagram of the continuous-flow experimental set-up is shown in Figure 3. Fresh seawater sampled near Hachijyo Island in the Pacific Ocean was first filtered to remove particulates larger than 10×10^{-6} m and then passed upward through the packed-bed adsorption column. The temperature of the seawater was controlled at 298 K using a heat exchanger. A bundle of 3,000 AO-H fibers, 15 cm in length, was charged in a 3.1 cm ID column that was 15 cm in height. The connection of two columns afforded a 30 cm high column. Seawater flow rates were

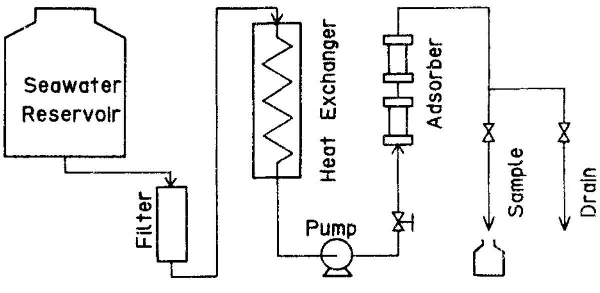

Figure 3. Apparatus for uranium adsorption.

controlled precisely for prolonged periods of time. Since the AO-H fibers function only as adsorbents, not as filtration membranes, seawater does not permeate through the AO-H fibers. The cumulative volume of seawater flowing through the column was recorded. The superficial velocity varied from 0.25 to 1 cm/s, resulting in 30 to 120 s contact times. After specific time intervals, 2 L of the seawater that had passed through the column was sampled. The uranium concentration of the sample seawater was determined according to an established method (Shijo and Sakai, 1982). The amount of uranium adsorbed on the adsorbent, averaged over the column, was calculated from the following equation,

$$\Delta q = uS\Delta t(C_O - C_e)/W \tag{8}$$

where Δq is the increased amount of uranium adsorbed during the time increment Δt. u, S, and W are respectively the superficial velocity of seawater, the cross-sectional area of the column, and the total weight of the AO-H fibers in the column. The pressure drop across the column was measured with a conventional manometer.

Elution

The bundle of AO-H fibers was taken from the bed after 24 days contact with seawater. To understand the distribution of uranium along the bed, the AO-H fibers were cut into 10 pieces in the direction of the bed height. To elute the uranium adsorbed quantitatively with hydrochloric acid, a concentration of more than 1M is necessary (Saito et al., 1987). The elution on each piece was achieved by overnight shaking with three successive portions of 1M hydrochloric acid. The uranium content in the eluates was usually determined colorimetrically using Arsenazo III reagent (Motojima et al., 1969). Other metals were determined in the eluates by atomic absorption spectrophotometry. When calculating the average amounts of elements adsorbed on the AO-H fiber over the bed, the concentration factor CF may be defined as

$$CF = (\text{g/kg adsorbent})/(\text{g/L seawater}) \tag{9}$$

Results and Discussion

Adsorbent

To prepare the AO-H fiber with the best possible uptake properties, an optimized conversion of the cyano group into an amidoxime group was necessary along with balancing the amidoxime group against by-product (Hori et al., 1987). Since the amount of HCl adsorbed corresponds to the amount of the amidoxime group, the amidoximation was terminated after 10 h, which provided for the maximum amount of adsorbed HCl. The conversion of the grafted cyano group into the amidoxime group was 34%. The amount of amidoxime group, 8.3 mol/kg of base polymer, was almost comparable to that of a commercial ion-exchange resin.

The properties of the AO-H fiber are summarized in Table 4. Figure 4 shows the distribution of copper across the fully copper-loaded AO-H fiber measured by XMA. The uniform copper distribution corresponds to the uniform distribution of amidoxime groups throughout the membrane. Okamoto et al. (1985) prepared the fibrous amidoxime-group-containing resin by radiation-induced grafting polymerization of acrylonitrile in the vapor phase onto a nonporous tetrafluoroethylene-ethylene copolymer fiber. They reported the distribution of functional groups located on the surface. Uniform distribution of the amidoxime group on the AO-H fiber in Figure 4 is due to high porosity (70%) of the porous polyethylene hollow fiber used as the base polymer.

Table 4. Properties of AO-H Fiber

Degree of grafting	128%	
Amount of CN group after grafting	24.2 mol/kg base polymer	
Amount of AO group	8.3 mol/kg base polymer	
	Dimension	
	d_i	d_o
Base polymer	0.27 mm	0.38 mm
AO-H fiber	0.32 mm	0.46 mm
Density	0.49 kg resin (HCl)/L resin (KOH)	

Adsorption rate

Since the inner and outer diameters of the AO-H fiber treated with alkaline solution were 0.032 and 0.046 cm, respectively, the porosity of the bed and the specific surface area of the adsorption column were calculated to be 0.66 and 97 cm^2/cm^3, respectively, from the following equation,

$$\epsilon = 1 - (d_o^2 - d_i^2)N/d_c^2 \tag{10}$$

$$a_v = 4(d_o + d_i)N/d_c^2 \tag{11}$$

where d_c, d_i, d_o, and n are respectively the column diameter, the inner and outer diameter of the AO-H fiber, and the number of fibers packed in the column. The ratio of the porosity in the inner part to that of the outer of the AO-H fiber is 9/10.

Figure 5 shows a plot of the amount of uranium recovered as a function of both dm^3 of AO-H fiber in the KOH-treated form and kg in the HCl-adsorbed form vs. contact time. We have obtained a uranium adsorption rate of 0.66 mg U/g of AO-H fiber over 25 days for a superficial velocity of 1 cm/s. This is finite large when compared with the rate of adsorption by

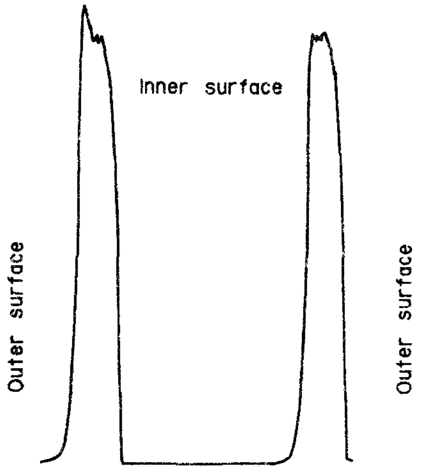

Figure 4. Distribution of Cu in AO-H fiber.

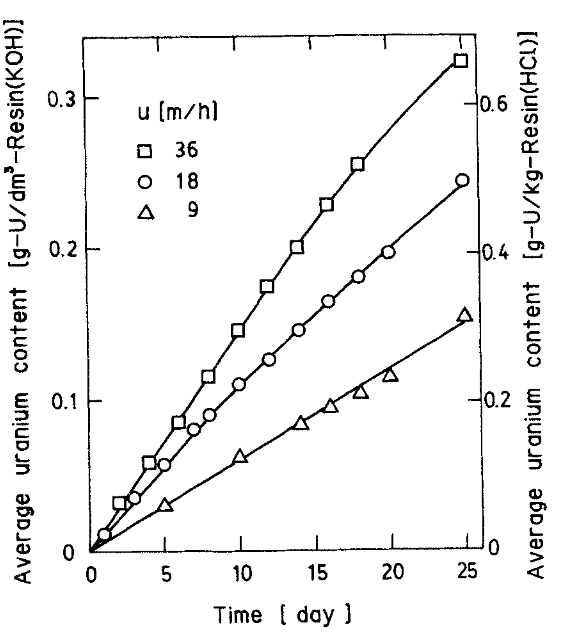

Figure 5. Uranium loading as a function of contact time.

hydrous titania granules, which has values of the order of 0.1 mg U/g of granules over 20 days. The value of 0.66 mg U/g, that is, 0.066%, is equivalent to the uranium content of low-grade uranium ore.

Selectivity

The distribution of uranium adsorbed along tne bed is shown in Figure 6 as a function of superficial velocity of seawater. At each superficial velocity, the sixth piece cut in the direction of the bed height had an amount higher than uranium adsorbed by the fourth and fifth pieces. Since the 30 cm long bed is made of two columns 15 cm long, the twist of the bundle of the AO-H fibers formed a dead region along the bed height, and mixing in the connection part resulted in an increase in the amount of uranium adsorbed at the sixth piece.

The concentration factors *CF* for some elements after 25 days exposure to seawater for a superficial velocity of 1 cm/s are shown in Table 5. The *CF* for uranium was found to be 200,000 and those of magnesium and calcium to be 3.3 and 6.3, respectively. These values are almost of the same order reported in our other amidoxime resins (Saito et al., 1987b; Uezu et al., 1987).

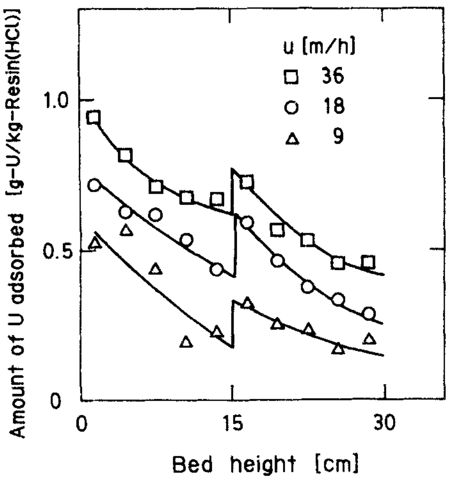

Figure 6. Distribution of amount of uranium adsorbed along the bed.

Table 5. Concentration Factors after 25 Day Contact, Superficial Velocity 1 cm/s

Metal	Conc. in Seawater g/L	Conc. Adsorbed g/kg	Conc. Factor L/kg
Uranium	0.0000033	0.66	200,000
Magnesium	1.4	4.6	3.3
Calcium	0.40	2.5	6.3

Flow resistance

The dependence of the pressure drop across the bed on the superficial velocity is shown in Figure 7. A superficial velocity of 1 cm/s gave a water head loss of about 14 cm. During the contact with filtered seawater over 25 days, the pressure drop was almost constant, and thus the blocking by bubbles and by solid precipitates evolved during the prolonged periods was found to be negligible. The novel packed bed suggested by this work is of greater advantage than conventional packed beds for stable operation.

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Notation

a_v = specific surface area, cm^2/cm^3
C_e = concentration of uranium at column exit, g U/cm^3
C_0 = concentration of uranium in seawater, g U/cm^3
D = diffusivity, cm^2/s
d_c = diameter of column, cm

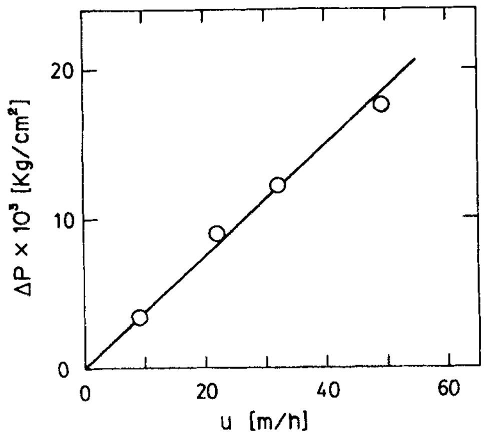

Figure 7. Pressure drop.

d_i = inner diameter of hollow fiber, cm
d_o = outer diameter of hollow fiber, cm
K_F = overall mass transfer coefficient, cm/s
k_f = film mass transfer coefficient, cm/s
L = length of column, cm
N = number of hollow fibers
n = constant in Freundlich isotherm equation
Q = dimensionless average resin-phase concentration
q_O = resin-phase concentration in equilibrium with C_O, g U/g
S = cross-sectional area of column, cm^2
t = time, s
U = dimensionless bulk concentration
U^* = dimensionless concentration in liquid in equilibrium with Q
u = superficial velocity of seawater, cm/s
W = total weight of AO-H fibers in column, g
x = dimensionless axial coordinate

Greek letters

ϵ = porosity
κ = defined in Eq. 4
ρ_b = bed density, g resin/cm^3 bed
τ = dimensionless time = $uC_Ot/(\rho_b q_O L)$

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