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Sangita Pal^a; V. Ramachandhran^a; S. Prabhakar^a; P. K. Tewari^a; M. Sudersanan^b

^a Desalination Division, Bhabha Atomic Research Center, Mumbai, India ^b Analytical Chemistry Division, Bhabha Atomic Research Center, Mumbai, India

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Polyhydroxamic Acid Sorbents for Uranium Recovery

SANGITA PAL,¹ V. RAMACHANDHRAN,¹ S. PRABHAKAR,¹
P. K. TEWARI,¹ AND M. SUDERSANAN²

¹Desalination Division, Bhabha Atomic Research Center, Mumbai, India

²Analytical Chemistry Division, Bhabha Atomic Research Center,
Mumbai, India

Crosslinked polyacrylamides were synthesized by solution polymerization using benzoyl peroxide as the radical initiator. The water-insoluble polymer obtained was functionalized by reacting with hydroxylamine to convert the amide group into hydroxamic acid group. The resultant functionalized polymer was characterized in terms of moisture uptake, elemental composition, IR spectra, thermal stability, exchange capacity and heavy metal sorption. The sorbent, obtained in particulate form, was investigated for its sorption properties with respect to uranium from uranyl nitrate solutions under unstirred batch conditions. This paper will concentrate on preparation, characterization and performance evaluation with respect to uranium sorption as a function of concentration, time, solution pH and temperature. The potential of this sorbent for uranium and other heavy metal ion recovery from sea water is ascertained.

Keywords uranium separation, crosslinked polyacrylamide, hydroxamic acid, sea water, solution polymerization

Introduction

Polymeric and copolymeric substrates are constantly being developed and used for complexation with metal ions either for ion exchange or selective adsorption. Removal of heavy metals from environmental samples is especially important because their excess intake leads to pronounced toxic symptoms. Uranium is one of the most seriously threatening heavy metal because of its toxicity and radioactivity. Extraction of uranium from seawater has also attracted attention the world over in view of the anticipated depletion of uranium resources in the near future (1–11). Though the concentration of uranium is extremely low, it constitutes a significant source due to the large quantity of seawater available. The adsorption method using solid adsorbents is promising for uranium recovery either from seawater or from uranium bearing effluents generated in the nuclear industry is promising with regard to economic and environmental impact. Among the inorganic sorbents, hydrous titanium oxide (HTO) has shown high selectivity

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Address correspondence to V. Ramachandhran, Desalination Division, Bhabha Atomic Research Center, Mumbai 400 085, India. Tel.: (+91) (022) (25594669); E-mail: ramanv25@yahoo.com

for uranium (2). Polymeric materials having, functional groups such as carboxylic, amide, nitrile, iminodiacetic acid, amidoxime, ammonium groups, etc. are hydrophilic, as well as exhibit ion exchange properties, which make them suitable for metal ion removal from aqueous solutions. Various polymeric adsorbents have been designed and successfully demonstrated for metal ion removal from dilute aqueous solutions. Resins based on polyamines, polyethyleneimines, polyallylamines, chelating resins functionalized with dithiocarbamate calixarene moiety functionalized on a polyethyleneimine support were all reported for uranium recovery. Resin fibers and fabrics containing amidoxime groups are extensively studied for uranium recovery from seawater. Omichi et al. used an amidoxime group containing polyacrylonitrile fibers for uranium recovery from seawater (12). Choi et al. (13–15) reported uranium adsorption using radiation induced grafting of polyethylene adsorbents carrying amidoxime and carbonyl groups. Amidoximation and characterization of N-vinyl-2-pyrrolidone/acrylonitrile systems are reported (16). Polyacrylamide-co-acrylic acid hydrogels prepared by gamma irradiation are used for uranium recovery (17). Amidoxime containing membranes and hollow fibers are used for uranium recovery from seawater (18), Egawa et al. (20) investigated uranium recovery from seawater on a amidoxime modified acrylonitrile divinylbenzene copolymer. Caykara et al. (21) reported uranium sorption from polyvinyl pyrrolidone grafted-g-citric acid hydrogels. Hydroxamic acids have long been known for their ability to complex heavy metals such as uranium and iron. They have a unique application as a protective coating for implantable medical devices or as a polymeric drug for the treatment of urinary stones associated with urea splitting bacteria. In addition, they show anticoagulant activity, and are also useful as ion exchanges (22). In this paper, a systematic study on the preparation, characterization and performance of variously cross-linked hydroxamic acids anchored onto an acrylic polymer backbone for metal ion removal, particularly uranium from dilute solutions is reported.

Experimental

Materials

Acrylamide procured locally, and N,N'-methylenebisacrylamide (E. Merck) were used as received without any purification. Preparation of crosslinked polyacrylamide and its conversion to hydroxamic acid are detailed below.

Preparation of Sorbents

Synthesis of Crosslinked Polyacrylamide (PAAM). Crosslinked polyacrylamides were prepared by polymerizing a solution of acrylamide and N,N'-methylenebisacrylamide in a different ratio with a fixed amount of initiator at 60°C. Gel formed after 20 min of reaction. After achieving room temperature, acetone was poured in the gel for phase inversion. After 24 h, the gel was crumpled and washed with water. After drying, it was crushed to reduce the particle size.

Conversion of PAAM to Polyacrylhydroxamic Acid Chelating Resin (PHOA)

A solution of a calculated amount of hydroxylamine hydrochloride and sodium hydroxide was added to a suspension of dry crosslinked PAAM. The resulting mixture was stirred for 5 min at room temperature. The reaction was continued for 6 h at 70°C. Ammonia was

liberated during the reaction. The resulting polymer was washed with: a) water, b) acidified with 3 (N) HCl solution and then again washed with, b) water till it is chlorine free. The reaction scheme (Scheme 1) is given below.

Physical and Chemical Characterization

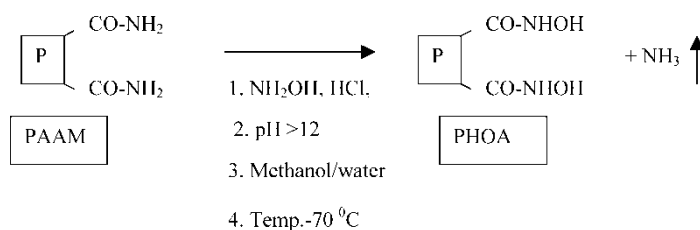
IR spectra of crosslinked polyacrylamide, polyhydroxamic acid and polyhydroxamic acid with sorbed uranium were taken using KBr pelletization. TGA spectra of samples were recorded in nitrogen atmosphere at a heating rate of 10°C/min. The ion exchange capacity was evaluated by equilibrating a known weight of the dry sorbent with 0.1 (N) HCl for 24 h, and the acid released estimated, when the sample was subsequently treated with sodium chloride solution. Batch experiments were carried out using solutions of various metal ion salts to study their uptake as a function of extent of cross-linking, time of contact, nature of metal ion, concentration and pH. In batch experiments, a known quantity of the sorbent was equilibrated with a definite volume of the salt solution in passive contact without any mechanical agitation. The sorbent, after a specified period of contact, was washed thoroughly and eluted using a 1 (N) HCl solution. The metal ions were analyzed by a Atomic Absorption Spectrophotometer. The uptake of metal ions by the sorbent was calculated according to the mass balance on the metal ions expressed as:

$$q = [(C_0 - C_e) \times V]/m \quad (1)$$

Here, q is the amount of metal ions sorbed on to the dry sorbent (mg/g); C_0 and C_e are the concentrations of the metal ions in the initial solution and in the aqueous phase at equilibrium, respectively (mg/L); V is the volume of the aqueous phase (L); and m is the mass of the dry sorbent used (g).

The metal ion loaded sorbent was placed in the elution medium and stirred for 1 h at room temperature. The elution volume was 50 mL. The elution ratio was calculated from the amount of metal ion sorbed on to the sorbent and the final concentration of metal ions in the elution medium by using the following expression:

$$\text{Elution ratio} = \frac{\text{Amount of metal ion eluted to the elution medium}}{\text{Amount of metal ions sorbed on to the sorbent}} \times 100$$



Where, P = the polymer backbone

Scheme 1. Conversion of PAAM to hydroxamic acid (PHOA) containing chelating resin.

Results and Discussion

Characterization

Three different polymer samples with varying acrylamide-N,N'-methylenebisacrylamide mole ratios, namely, 0.95/0.05, 0.85/0.15, 0.75/0.25 were synthesized, functionalized and designated as PHOA 9505, PHOA 8515 and PHOA 7525, respectively. Their characterization data in terms of elemental composition are given in Table 1 and ion exchange capacity in Table 2.

Comparison of calculated and observed elemental composition values indicate that the conversion of acrylamide to hydroxamic acid is close to 60%. The IR spectra recorded for the (a) polyacrylamide (PAAM), (b) poly-hydroxamic acid (PHOA) and, (c) uranium loaded PHOA samples are given in Figure 1. Characteristic absorption peaks for N-H and N-OH stretching at 3100 to 3400 cm^{-1} , carbonyl group stretching peak at 1600 cm^{-1} , N-H bending peaks at 1100 cm^{-1} and N-O stretching peak at 944 cm^{-1} are observed in all the samples. Comparison of spectra of the PHOA sample and uranium loaded PHOA samples show a shift in the N-O band at 944 cm^{-1} , initially observed in the first sample, to 880 cm^{-1} in the uranium loaded sample as a result of interaction of corresponding groups with UO_2^{2+} . This indicates a strong interaction of uranyl ions with the N-O group of the sorbent and as the M-O bond is stronger, the N-O bond exhibits lower stretching frequency. Additional peaks around 900 cm^{-1} can also be seen which are characteristic of O-U-O stretching vibrations.

Figure 2 shows TGA curves of PAAM 9505, PAAM 7525, PHOA 7525, arsenic complex of the same sorbent and uranium complex of the above polymer sorbent. Weight loss is observed at three different temperature regions for the unreacted cross-linked polyacrylamide, as well as polyhydroxamic acid derivatives. In the case of metal complexes, weight loss has been observed at two temperatures only. The first weight loss observed, a) at around 150°C, due to the loss of moisture from the sorbent, the second weight loss observed, b) at around 250°C, as due to the imidization reaction and

Table 1
Physicochemical properties of polyhydroxamic acid sorbent

S.no.	Polymer	Elemental composition (wt%)			
		%C	%H	%N	%O
1	PHOA 9505	Obsd. 44.12	6.41	17.69	31.78
		Calcd. ^a 41.65	5.12	15.95	36.48
		Calcd. ^b 44.93	6.42	17.20	31.45
2	PHOA 8515	Obsd. 45.09	6.82	17.65	30.44
		Calcd. ^a 40.29	5.90	16.38	37.43
		Calcd. ^b 43.86	6.42	17.83	31.89
3	PHOA 7525	Obsd. 43.28	6.47	17.27	31.98
		Calcd. ^a 39.7	5.98	16.54	37.78
		Calcd. ^b 42.9	6.44	17.89	32.77

^aCalculated based on 100% conversion.

^bCalculated based on 60% conversion.

Table 2
Metal ion hydrogen exchange capacities

S. no.	Metal ion	Exchange capacity (m · mol./g)		
		PHOA 9505	PHOA 8515	PHOA 7525
1	Sodium-hydrogen exchange	1.6	1.04	0.96
2	Copper-hydrogen exchange at pH 3	1.9	1.05	1.01
3	Iron-hydrogen exchange at pH 1	0.62	0.49	0.41

the third weight loss observed, c) at around 400°C, as due to the elimination of the pendant functional group. The extent of moisture loss is observed to be lower in the case of the hydroxamic acid derivative, probably due to enhanced intermolecular or intramolecular interaction leading to lower moisture uptake. Moisture loss and imidization get shifted to slightly higher temperatures with increased crosslinking. In the case of metal complexes, the moisture loss is nearly absent and the imidization peak is shifted to higher temperatures. This clearly suggests that the imidization reaction is difficult due to complex formation.

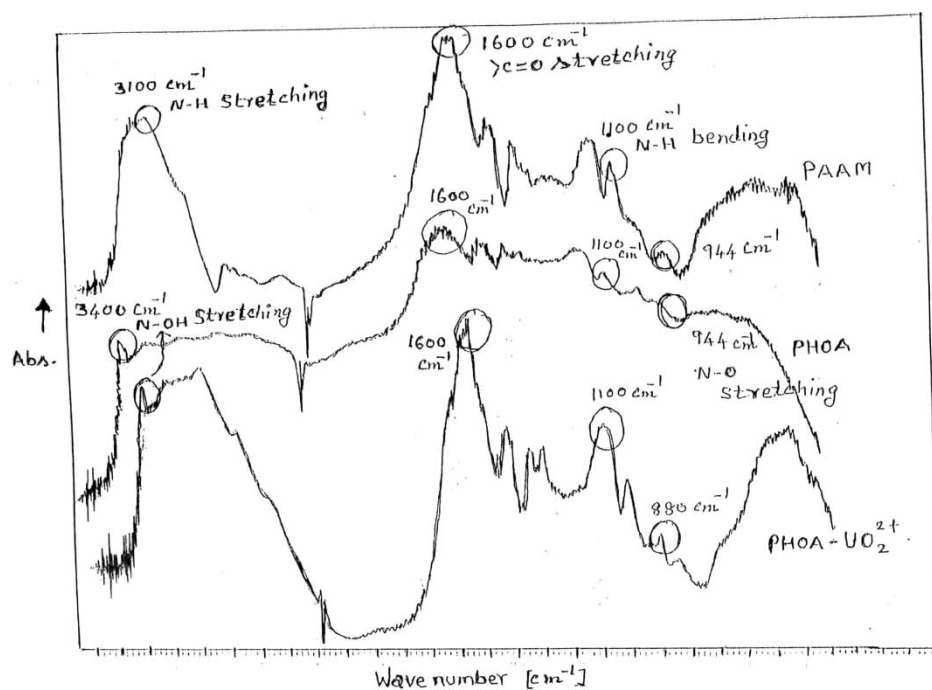


Figure 1. FTIR spectra of PAAM, PHOA and metal ion-PHOA complex.

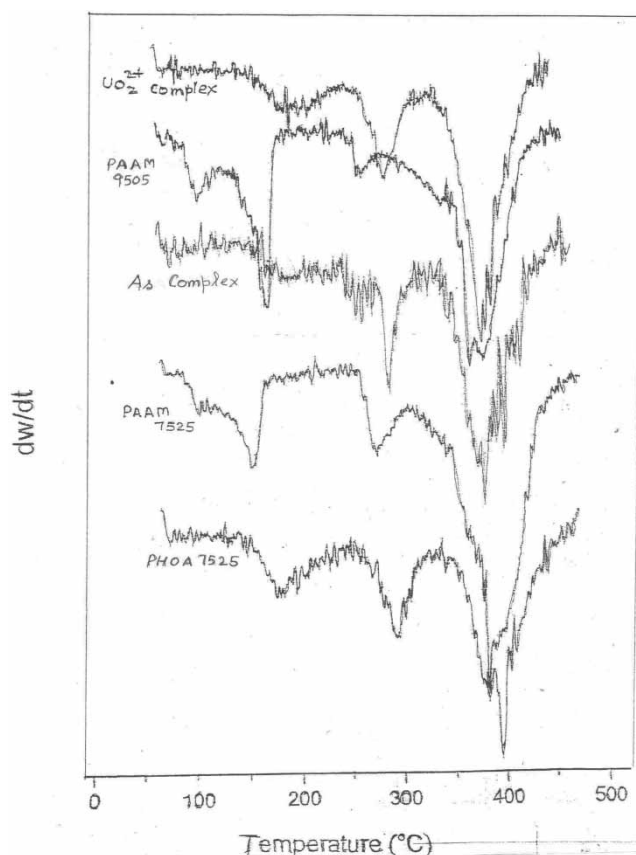


Figure 2. TGA spectra of polyacrylamide, polyhydroamic acid and metal absorbed sorbents.

The moisture uptake as a function of relative humidity is given in Figure 3. It can be seen that the moisture recovery is reduced with a increasing crosslinking and lower humidity. For example, PHOA 9505 derivative picks up 29% moisture at 90% relative humidity (RH) and 5.3% moisture at 29% RH. PHOA 7525 crosslinked derivative picks up only 2.8% moisture at 29% RH. Comparison of the unreacted polyacrylamide with its hydroxamic acid derivative indicates that the unreacted polymer picks up more moisture, probably due to increased intermolecular or intra molecular bonding in the case of the reacted derivative. This is also indicated in the respective TGA curve in Figure 2. Higher moisture uptake may allow better interaction with metal ions. The degree of crosslinking affected the moisture uptake of the polymer, as well as decreasing the concentration of free amide groups available for conversion.

Metal-ion exchange capacities for the hydroxamic acid derivatives are obtained for various metal ions namely; sodium, copper, and iron and the values obtained are given in Table 2. It can be seen that the metal ion hydrogen exchange capacity depends on the nature of the metal ion with highest exchange capacities observed for sodium and copper.

Upon addition of a Cu(II) or Fe(III) solution to the fully hydrated polymer, the polymer turns dark green or deep maroon, respectively. The color change is almost instantaneous for the lower crosslinked polymer and it intensifies with an increase in

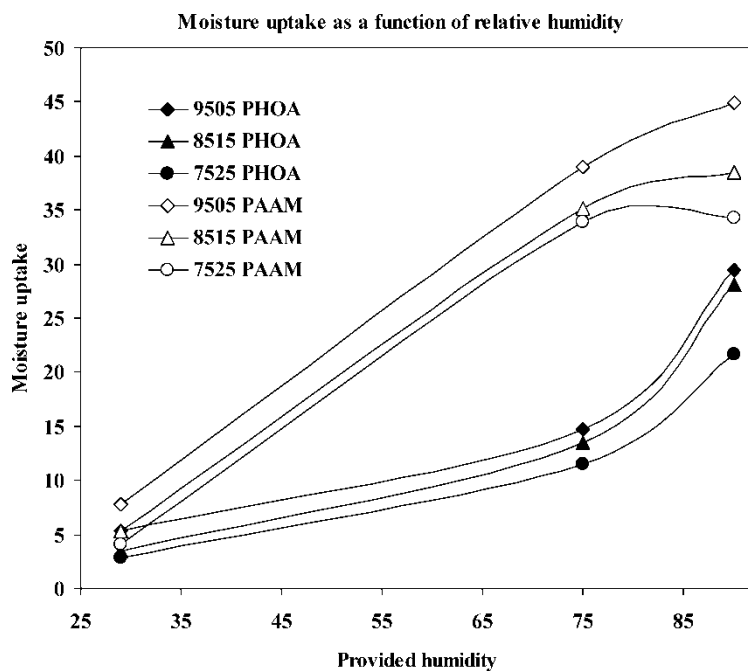


Figure 3. Moisture uptake as a function of relative humidity: ◆ 9505 PHOA, ● 8515 PHOA, ▲ 7525 PHOA, ◇ 9505 PAAM, ○ 8515 PAAM, △ 7525 PAAM.

concentration of metal ion. In the case of sodium, no color change was noticed. In the case of PAAM, the original color of the polymer is retained when contacted with metal ions, indicating no significant interaction in comparison.

Initial pH of the equilibrating metal ion solution critically influences both the sorbent surface chemistry, as well as the solution chemistry of the dissolved metal ions. The iron(III), UO_2^{2+} and copper(II) uptakes by the polymer at various pH are given in Table 3. The metal binding is pH dependent. At very low pH, copper uptake is very low, whereas iron and uranium uptake is consistent. An increase in pH increases metal ion uptake. At lower pH, hydrogen ions may be competing with the metal ions and depending on their affinities for the donor groups, the pH effect on uptake is either significant or marginal. Since copper uptake is low at low pH as compared to uranium and iron, the latter appears to have relatively stronger affinities relative to hydrogen ions than the former. The differences in metal uptake between copper and iron at low pH could be useful in their separation at pH 1 or lower. Uranium uptake is also found to be strongly pH dependent. At lower and higher pH values, the uptake is found to be low. The reduced uptake at low pH values could be due to the possible protonation of the carbonyl groups. In comparison to styrene-acrylonitrile-amidoxime sorbent reported previously from this laboratory (23), the crosslinked polyacrylamide hydroxamic acid shows much higher uranium uptake, probably due to the increased hydrophilic character of the present sorbent.

The uptake of various metal ions namely, nickel, cobalt, zinc, uranium, copper and iron are presented in Table 4 with their characteristic color change noticed in the polymer sample (PHOA 9505 and 8515) subsequent to metal ion loading. The metal

Table 3
pH Dependence of copper(II) Iron(III) and UO_2^{2+} uptake

S. no.	Acidity of metal ion solution	Uptake (mg/g)		
		PHOA 9505	PHOA 8515	PHOA 7525
1	1 Cu(II)	6.2	4.3	2.8
2	3 Cu(II)	49.1	31.5	27.7
3	5 Cu(II)	54.9	34.6	30.4
4	1 Fe(III)	33.4	30.7	26.1
5	3 Fe(III)	34.9	31.3	27.5
6	5 Fe(III)	36.2	33.5	29.4
7	1 UO_2^{2+}	52.9	49.1	44.3
8	3 UO_2^{2+}	87.05	79.4	69.4
9	5 UO_2^{2+}	94.6	90.6	83.2
10	7 UO_2^{2+}	75.2	71.9	63.2
11	8 UO_2^{2+}	61.3	55.1	42.9

Sorbent: 0.5 g.

Metal ion concentration (initial): 1%.

Volume of equilibrating solution: 50 mL; Time of contact: 6 h.

ion uptakes for the various samples are in the order of their hydrophilic character. Higher moisture uptake of the sorbent shows higher metal ion uptake.

The order of metal ion affinity for the donor groups is found to be $\text{Fe(III)} > \text{Cu(II)} > \text{UO}_2^{2+} > \text{Zn(II)} > \text{Ni(II)} > \text{HAsO}_4(\text{II}) > \text{Co(II)}$. The ease of elution of the sorbed metal ion also was found to be $\text{Co(II)} > \text{Ni(II)} > \text{Cu(II)} > \text{Zn(II)} > \text{HAsO}_4(\text{II}) > \text{UO}_2(\text{II}) > \text{Fe(III)}$. Iron was found to be the most difficult element to get eluted and the elution

Table 4
Uptake of various metal ions and color change on polymer sorbent

S. no.	Metal ion	Uptake	Elution	Uptake	Elution	Colour change on polymer
		(mg/g) PHOA 9505	ratio (%)	(mg/g) PHOA 8505	ratio (%)	
1	Ni(II)	10.2	91.6	8.4	86.2	Light parrot green
2	Co(II)	6.9	98.4	5.7	94.5	Reddish pink
3	Zn(II)	15.7	87.1	13.6	81.7	Pinkish white
4	UO_2^{2+}	79.4	81.5	71.5	76.9	Deep orange
5	Cu(II)	34.6	79.4	31.9	70.4	Deep green
6	Fe(III)	33.5	64.3	24.7	60.5	Deep maroon
7	$\text{HAsO}_4(\text{II})$	19.6	95.7	16.8	91.2	Deep reddish orange

Sorbent: 0.5 g.

Metal ion concentration (initial): 1%.

Volume of equilibrating solution: 50 mL; Time of contact: 6 h.

ratio improves with an increase in acid strength. Elution depends on, a) the extent of hydration of the metal ion, b) polymer microstructure and, c) the binding strength between the metal ion and the functional group.

The uptake of UO_2^{2+} as a function of initial concentration is shown in Figure 4. The uptake value increases with an increase in initial concentration and a saturation value is achieved at ion concentration of 3500 mg/g, which represents saturation of the active binding sites on the sorbent. The hydroxamic acid content of the sorbent used in this experiment was 13.9 m. mols/g. The maximum UO_2^{2+} sorption obtained is 0.40 m · mol per unit mass of the sorbent. The low stoichiometry of sorbed uranyl ion against available hydroxamic acid groups suggests that all the functional groups are not accessible for chelation.

Figure 5 shows the effect of time on the UO_2^{2+} uptake for the PHOA 9505 sample for different concentrations. The rate of uptake was observed to be sufficiently fast to reach equilibrium in about 100 min. The uranium uptake is higher for higher initial concentration, but the rate of uptake is similar for the concentration range studied. Reported experimental data on sorption kinetics of uranium on polymeric adsorbents have shown a wide range of values. Omichi et al. (12) reported more than 7 days time to attain equilibrium for uranium separation from seawater with amidoxime containing polyacrylonitrile fibers. Denizli et al. (24) have reported nearly 100 mins equilibration time for uranium adsorption with methacryloylamidoglutamic acid functionalized poly hydroxyethyl methacrylate, which matched with the values obtained in our studies.

The effect of temperature of uranyl nitrate solution on uranium uptake by PHOA 9505 sample was evaluated in the temperature range of 5°C to 50°C for 1000 ppm uranyl nitrate solution for 6 h contact time. The results are given in Table 5. It can be seen that uranium

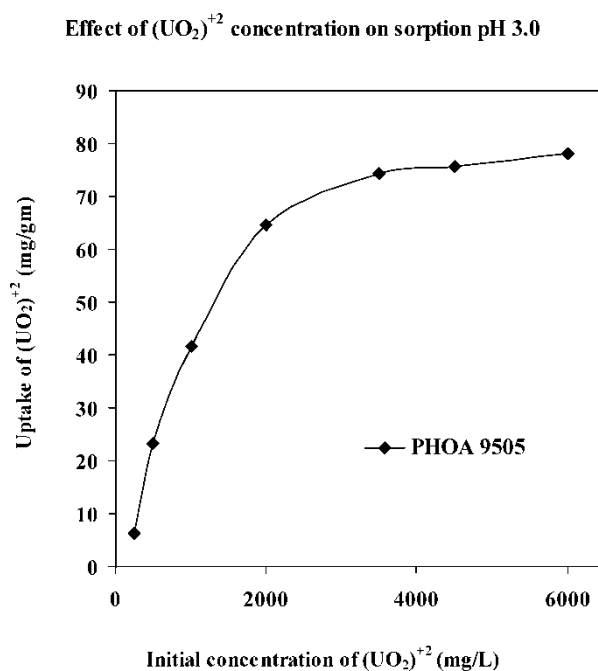
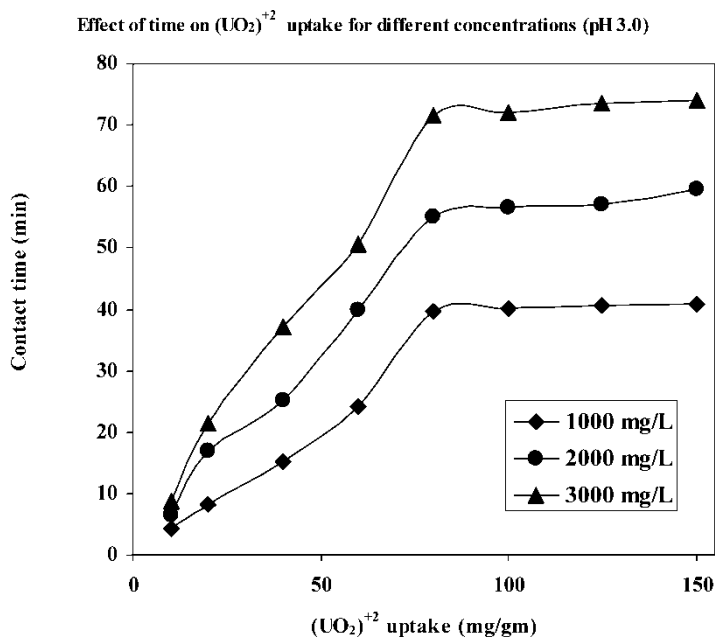


Figure 4. Effect of $(\text{UO}_2)^{2+}$ concentration on sorption at pH 3.0 ◆ 9505.



Sorbent : 0.58 PHOA 9505
Volume of equilibrating solutions : 50 ml

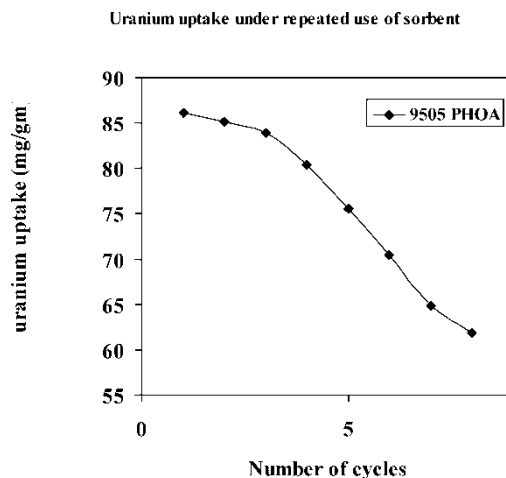
Figure 5. Effect of time on UO_2^{2+} uptake for different concentration at pH 3.0. \blacklozenge 1000 mg/L, \bullet 2000 mg/L, \blacktriangle 3000 mg/L.

uptake increases in temperature from 56.5 mg/g to 120.2 mg/g when the temperature is raised from 5°C to 50°C. The effect of increasing temperature changes the morphology of the sorbent, enhances the diffusion of the metal ion inside the pores of the sorbent and provides the required binding energy for uptake. The morphology changes could be the controlled shrinking/swelling of the matrix due to the presence of a crosslinking agent affecting the diffusion path length and free volume inside the pores. The observed increase in uptake, could be attributed to the combined effect of all the above.

Table 5
Uranium uptake as a function of temperature

S. no.	Temperature (°C)	UO_2^{2+} uptake (mg/g)
1	5	56.5
2	15	69.7
3	27	87.1
4	50	120.2

Sorbent: 0.5 g.
Metal ion concentration (initial): 1%.
Volume of equilibrating solution: 50 mL; Time of contact: 6 h.



Sorbent : 0.5 gm PHOA 9505
 Equilibrating Solutions : 50 ml of 1% uranyl nitrate
 Contact time : 6 hrs

Figure 6. Uranium uptake under repeated use of sorbent ◆ 9505. Sorbent:- 0.5 gm PHOA 9505, Equilibrating solutions: 50 ml of 1% uranyl nitrate, Contact time: 6 h.

The repeated usability of the sorbent was evaluated by collecting uranium uptake data with the same sample PHOA 9505 under identical conditions of sorption-elution cycles. The results are given in Figure 6. It can be seen that the uranium uptake has declined from 86.1 mg/g to 61.8 mg/g after 8 cycles.

Recovery of uranium and other trace heavy metals from seawater by the PHOA 9505 sample was evaluated under column conditions with 50 liters of filtered sea water passing through a glass column containing 1 g of the sample. The sorbed titanium, vanadium, uranium, cobalt, and molybdenum were analyzed. The uptake of various heavy metals and their concentration factors are given in Table 6. The concentration factor (L/g) was worked out from the ratio of concentration of heavy metals sorbed ($\mu\text{g/g}$) to that present in seawater ($\mu\text{g/L}$). It can be seen that the sorbent exhibits high selectivity not

Table 6
 Recovery of uranium and other trace heavy metals from seawater

S. no.	Element	Conc. in seawater ($\mu\text{g/L}$)	Uptake ($\mu\text{g/g}$)	Concentration factor (L/g)
1	Ti	1	0.18	0.18
2	U	3.3	18.2	5.5
3	V	1.9	6.0	3.16
4	Co	0.4	5.6	14
5	Mo	10	1.13	0.113

Sorbent: 1 g PHOA9505.
 Adsorbate: 50 L of seawater.
 Average flow rate: 3 mL/min.

only for uranium, but also for cobalt and vanadium as well. The selectivity for molybdenum and titanium are found to be poor.

Conclusions

Insoluble polyacrylamide-hydroxamic acid sorbents with varying crosslinking were synthesized in the laboratory using benzoyl peroxide as the radical initiator. Their metal ion sorption characteristics were evaluated for copper, iron, uranium, zinc, arsenic, nickel, cobalt and uranium. The uranium uptake by the sorbent was studied in detail for various concentrations, pH, time of contact and temperature. The uranium uptake is higher for higher concentrations and is optimum at pH 6. Higher temperature improves the uranium uptake. For a given contact time, the rate of uptake is faster as compared to amidoxime based sorbent studied and reported earlier from this laboratory. The sorbent is reusable but repeated use reduces the sorption capacity. The sorbent selectively picks up uranium, vanadium and cobalt from natural seawater.

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