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Removal of uranium (VI) from liquid waste of calcareous shale, Allouga, southwestern Sinai, Egypt

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ABSTRACT

One of the main problems faced at the Allouga uranium project during the recovery of uranium was the presence of high chloride content in the uranium mineralization that ranged between 6 and 8%. It was necessary to remove the chloride by washing the uranium-bearing material several times with freshwater. This procedure used a large amount of water and the washed pregnant solution contained higher than 12 g/l of chloride and 10 mg/l of U(VI). The obtained solution had to be kept in vats and tanks which presented environmental problems due to the inability of quaternary amine resins such as D263B (Chinese resin which is considered to be the most suitable tool for uranium recovery) to extract the solution containing high chloride. Synthetic acrylamidoxime and hydrazinyl amine resins were used on polyethylene non-woven fibers, followed by Fourier transform infrared spectroscopy, SEM, and elemental analysis. The synthetic resins have a chelating site for the UO_2^{2+} and exhibited a very high capacity for metal sorption. It seems that this synthetic resin has 1,000 times higher rate of adsorption than D263B. This was due to its high affinity for U (VI) and low affinity for chloride.

Keywords: Allouga; Calcareous shale; Uranium; Waste; Resin

1. Introduction

It is inevitable that in the next 100 years power will be mostly generated from nuclear energy by utilizing uranium and most of the higher grade uranium will be exhausted; therefore, demand for lower grade uranium in either ores or solutions will increase [1–3].

The most common methods for uranium recovery are: (i) direct chemical methods, (ii) electrochemical treatments, (iii) ion exchange, and (iv) biosorption methods. Most of these methods have not been used in the treatment of dilute concentrations because they are expensive and have bad effect on the environment [4–6]. Ion-exchange adsorption method, especially the one that uses quaternary amine resins (QA), is well known to be the most suitable tool for uranium extraction due to its availability, reusability [7], speed, ease of extraction, and inexpensiveness, and so is used in most uranium projects [8–15].

High chloride concentrations of more than 2 g/l lead to not only competition for uranyl adsorption on QA resin but also partial elution of adsorbed ions [7]. The presence of chloride in ore depends on its mineral constituents and, consequently, shows up in leach liquor after chemical process (acid or alkaline leaching). The Allouga mineralization contains high chloride percentage (up to 7%); therefore, the pregnant solution was not suitable for adsorption on QA resin as D263B by direct leaching. It has been proven in several studies that the best and least expensive way to remove or reduce the chloride from ore minerals was washing with water and making it ready for subsequent chemical recovery process. The time of washing process depends on the original chloride concentration, ore grain size, and contact time [16].

The prepared washing solution was uranium contaminant (higher than 10 mg/l) and had high chloride (higher than 11 g/l). This makes it impossible to recover the uranium by QA resin.

This paper deals with the current state of uranium collection technology including development of adsorbent for uranium collection from washing waste solution. This is accomplished by the use of prepared organic adsorbents that are very selective for uranium adsorption and have a high surface area (coating on polyethylene non-woven fiber).

2. Experimental

2.1. Specification of the mineralized sample

The lithology of interest in Allouga, at Abu Zenima, is the Um Bogma Formation which is shown in Fig. 1. The formation carries a suite of poly-metallic elements, including secondary uranium, weathered copper sulfides, manganese, and iron oxides [17]. It also carries carbonates, such as calcites and dolomites, and sulfates in the form of gypsum and spangolite. The primary uranium minerals are coffinite and brannerite, while the secondary minerals are autunite and uranophane [17].

2.2. Mineralization characteristics

Table 1 shows the complete chemical analysis of the mineralized sample. Major oxides were analyzed by the use of rapid silicate analysis method [18]. Copper, zinc, cobalt, and nickel were analyzed by atomic

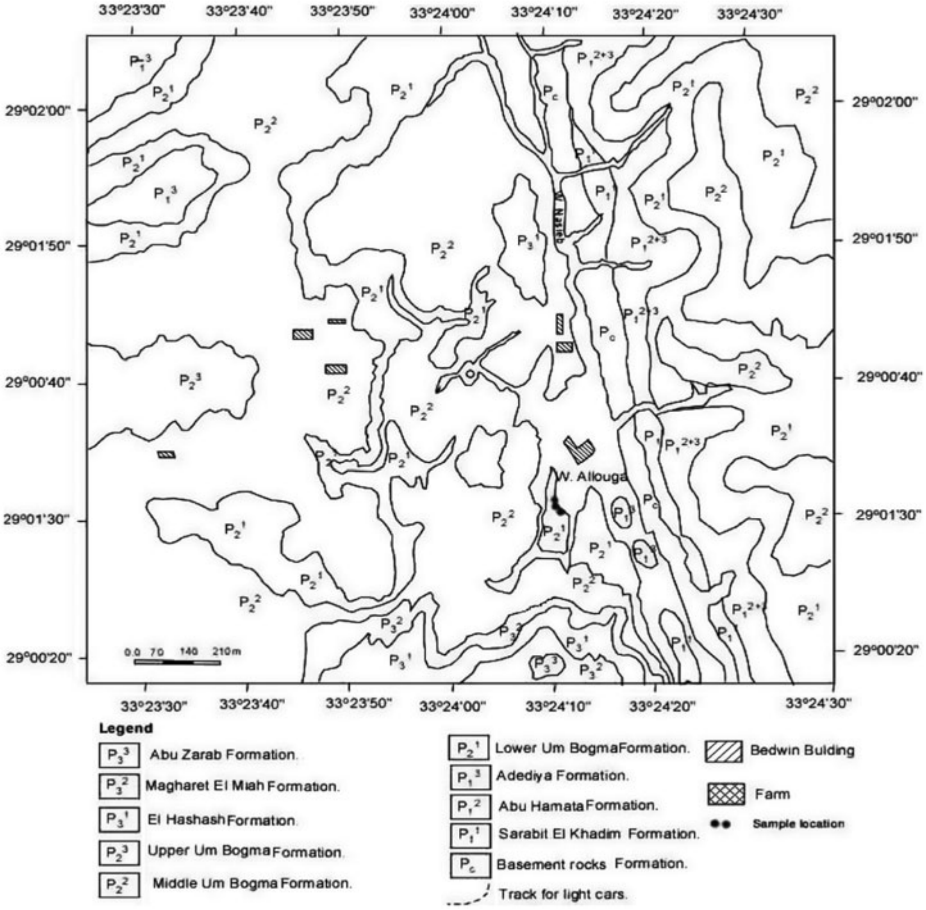

Fig. 1. Geological and location maps of the studied area.

Table 1
Element oxides (%) of Allouga materialization

Element oxide	%	Element oxide	%	Element oxide	%
SiO_2	26.18	K_2O	0.82	U_3O_8	0.029
Al_2O_3	13.01	ZnO	1.92	P_2O_5	1.05
Fe_2O_3	8.2	CuO	0.21	CO_3	3.9
MnO_2	0.91	TiO_2	0.14	Cl^-	4.6
CaO	15.55	NiO	0.84	SO_4^{2-}	2.72
MgO	3.06	CoO	0.013	LOI	15.60
Na_2O	1.14				

absorption method using a Unicam Atomic Absorption Spectrometer (model 969) at the following wavelengths: 222.60, 307.60, 243.70, and 305.10 nm, respectively [19]. Aluminum and manganese were determined using AAS at wavelengths 309.30 and 403.10 nm, respectively [20]. Uranium (VI) was determined using an oxidimetric titration method against ammonium metavanadate with diphenylamine sulfonate as indicator. Prior to titration, proper reduction of U(VI) was performed using ammonium ferrous sulfate [21]. Low concentrations of uranium (under 10 ppm) were determined using ICP-MS (VG PQ Ex Cell, Thermo Elemental). Chloride was determined by titration against silver nitrate as in Table 1 [22], and the chloride, sulfate, and carbonate contents were high (4.6, 2.72, and 3.9%, respectively). The analyzed sample had 26.18% SiO_2, 13.01% Al_2O_3, 15.55% CaO, 0.91% MnO_2, 0.84% NiO, 1.92% ZnO, 8.2% Fe_2O_3, 0.21% CuO, 0.14% TiO, 0.013% CoO, 0.0290% U_3O_8, and 0.001% lanthanides.

2.3. Method for preparation of grafted resins

The purpose of the present study was to investigate the possibility of recovering uranium from solutions with high chloride content. The prepared resins contained active sites that were unaffected by high chloride concentration, had hydrophilic properties, were basic in character (pH of washed pregnant solution [WPS] was 7.2), and were capable of adsorbing ions in either acidic or basic medium. The resin had relatively large surface area that could easily diffuse the ions inside the pores and reached saturation in a short period of time.

2.3.1. Synthesis of grafted inert polymer

About 0.1 g of asobisisobutyronitrile was dissolved in a mixture of 9.5 g acrylonitrile and 0.5 g divinyl benzene (DVB); 24.2 g of methylbenzoate, 20 vol.%, and dioctylphthalate, 80 vol.%, were added dropwise with strong agitation, and then 73.0 ml of 1% polyvinyl alcohol was added. A stream of nitrogen gas was bubbled in the mixture and then poured on polyethylene fiber (PE). The solution was heated at 65˚C for 6 h and the coated fiber was filtered off, washed with methanol, and dried at 50˚C for 4 h to produce grafted polyacrylonitrile divinylbenzene resin (GPAN) (Fig. 2).

2.3.2. Synthesis of grafted polyacrylamidoxime resins

Twenty grams of hydroxyl amine hydrochloride was dissolved in 15 ml methanolic solution (methanol: water 5:1), and then adjusting to pH 10 by NaOH. The solution was poured over 10 g of GPAN. The reaction was carried out for 2 h at 70˚C to yield corresponding grafted polyacrylamidoxime resin (GPAm) as in Fig. 3 [23–26]. GPAm was filtered, washed several times with methanolic solution, and dried for 5 h at 50˚C.

2.3.3. Synthesis of grafted polyhydrazinyl amine resin (GPHA)

A mixture of 30 ml hydrazine hydrate 99% and 32 ml ethanol was added to 10 g of GPAN and refluxed for 5 h, then filtered, washed several times with ethanol, and dried for 4 h at 50˚C, yielding grafted polyhydrazinyl amine resin (GPHA) as shown in Fig. 4 [27].

2.4. Characterization of synthesized resins

2.4.1. Elemental analysis

The elemental analysis of the resins was performed using a Perkin–Elmer 240 C Elemental Analytical Instrument. The oxygen, nitrogen, carbon, and

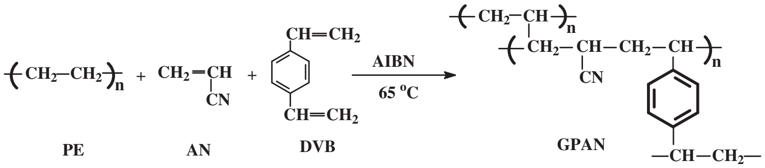

Fig. 2. Preparation of GPAN. *Conditions:* monomer-to-fiber ratio 4:1, cross-linker-type DVB, cross-linking ratio 5%, temperature 65˚C, and reaction period 6 h, under nitrogen.

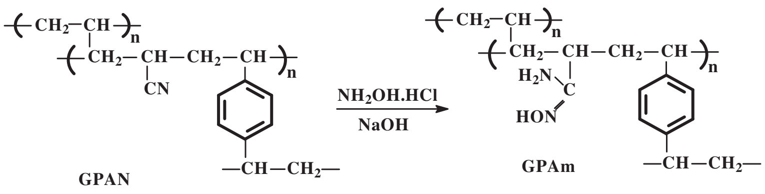

Fig. 3. Synthetic scheme of grafted acrylamidoxime polymer. *Conditions:* temperature 70˚C, reaction period 2 h.

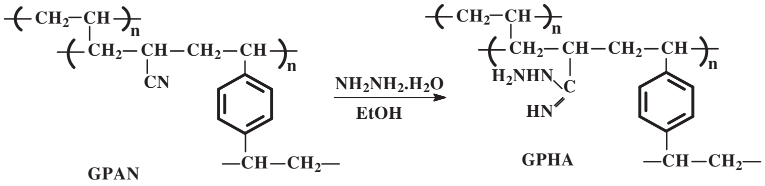

Fig. 4. Synthetic scheme of GPHA resin. *Conditions:* temperature 50˚C, reaction period 5 h.

hydrogen contents of GPAm were about 12.2, 25.4, 54.6, and 7.8%, respectively, whereas the nitrogen, carbon, and hydrogen contents of GPHA were about 35.2, 55.9, and 8.9%, respectively.

2.4.2. *FT-IR spectroscopy*

Fourier transform infrared (FT-IR) of the prepared resins was performed by JASCO 460 plus FT-IR spectroscopy model. The FT-IR spectroscopy for GPAN showed a peak at 2,990–2,970 cm^{-1} for C–H aliphatic stretch, a sharp peak at 2,240 cm^{-1} for CN stretch, and peaks at 1,485 and 1,079 cm^{-1} for CH_2 bend and C–N stretch, respectively. For GPAm, CN peak completely disappeared and a new broadband appeared at 3,350 for N–H and O–H stretch, peaks at 2,980, 1651.8, 1,115, and 79 cm^{-1} for C=N, CH aliphatic, C–N stretch, and N–H bending, respectively.

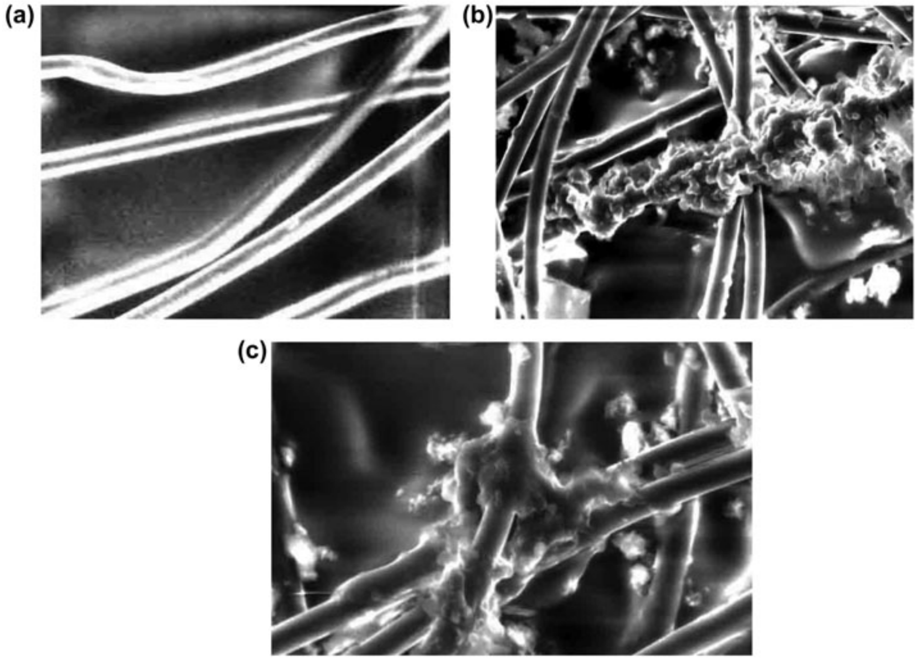

Fig. 5. SEM images of the prepared resins: (a) PE non-woven fiber before coating, (b) coated GPAm resin on PE fiber, and (c) coated GPHA resin on PE fiber.

For GPHA resin, peaks at 1640.4 cm^{-1} for C=N, broadband at 3,350 for N–H and NH_2 stretch, peaks at 2,960, 1,100, and 777 cm^{-1} for C–H aliphatic, C–N stretch, and N–H bending, respectively, appeared. Small shifts were present in loaded resins due to the effect of metal ions, indicating that no chemical changes had occurred.

2.4.3. SEM analysis

The structural morphology of the resin's surface was determined by SEM, model JEOL_JSM_5400 scanning microscope, shown in Fig. 5(a)–(c), which shows a coating of polymer on the fiber, which affected its swelling and metal uptake properties.

2.4.4. Grafting degree

The ratio of the coating polymer on the fiber, known as grafting degree (Gd%), was calculated using Eq. (1).

$$\mathrm{Gd}\% = (W_f - W_o) \times 100 \times W_f^{-1} \quad (1)$$

where Gd % is the grafting degree, and W_o and W_f are the weight (g) of fiber before and after coating, respectively. Grafting degrees for GPAm and GPHA were 92 and 90%, respectively.

2.4.5. Soluble fraction

The fractional loss in weight of the inert polymer is known as soluble fraction (SF%). It was determined via soxhlet extraction technique using dimethyl formamid at 200°C and calculated by Eq. (2) [26–29].

$$\mathrm{SF}\% = (W_f - W) \times W_f^{-1} \times 100 \quad (2)$$

where SF% is the soluble fraction, and W_f and W are the weight of the disks before and after extraction, respectively. The SF % was determined to be 3.3 and 3.5% for GPAm and GPHA, respectively [28].

2.4.6. Swelling properties

The maximum swelling time in deionized water and 0.1 N NaCl was 13 and 15 min for GPAm, respectively, and 15 and 18 min for GPHA, respectively. Swelling time for GPAm was shorter than that of GPHA. This was due to high hydrophilicty of GPAm. Swelling ratio in water and salt solution for GPAm was 33 and 27.5%, respectively, whereas, for GPHA was 30 and 25%, respectively. Swelling in salt solution was less than in water and took longer time due to "charge screening effects" of Na^+ ions which shielded the functional groups and prevented the effect of anion–anion repulsion [28].

2.5. Characterization of U(VI) complexes

Uranium has a high tendency to form complexes compared to other metals depending on pH values [15]. At lower pHs, uranium dissolves as UO_2^{2+}, which has two sulfate anion derivatives e.g. $UO_2(SO_4)_2^{2-}$ and $UO_2(SO_4)_3^{4-}$. In a carbonate media, a series of strong anionic aqueous carbonate complexes are present

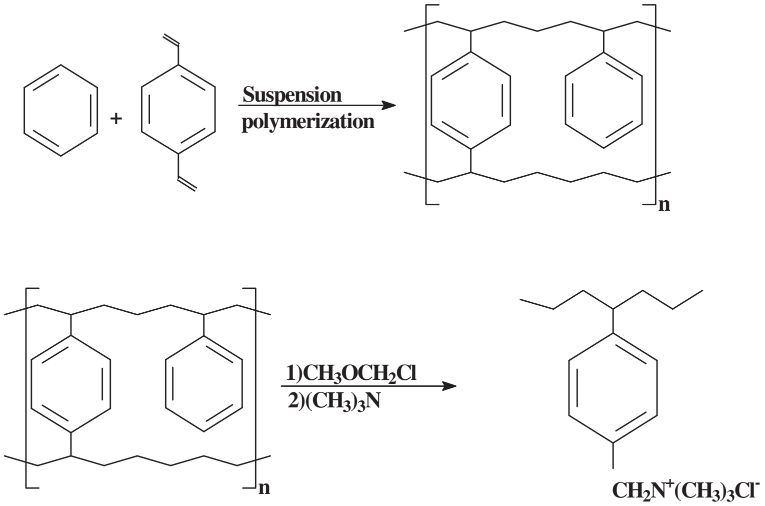

Fig. 6. General method for synthesizing quaternary ammonium D263B resin. *Conditions*: cross-linker-type DVB, cross-linking ratio 15%.

e.g. [$UO_2CO_3^0(aq)$, $UO_2(CO_3)_2^{2-}$, $UO_2(CO_3)_3^{4-}$, and $(UO_2)_3(CO_3)_6^{6-}$]. $UO_2(CO_3)_2^{2-}$ is the predominant form of uranium between pH 6 and 7 [30].

2.6. Characterization of D263B resins

D263B resin (QA resin used for uranium recovery at Allouga) was obtained from Borun Chemical Co. Ltd (China). It was styrene DVB of quaternary ammonium group. It is a strongly basic anion-type, semi-rigid gel prepared as spherical beads, is highly ionizable, usable over a large pH range, stable in the absence of strong reducing or oxidizing agents, insoluble in most common solvents, can withstand temperatures of up to 60℃, and has fast elution rate [31]. Fig. 6 shows the procedure for synthesizing the QA resin.

Uranyl carbonate as well as uranyl sulfate were adsorbed on QA resins to yield quaternary ammonium uranyl sulfate and carbonate complexes $(RN)_4UO_2(X_2)_3$ and $(RN)_2UO_2(X_2)_2$ (where RN = quaternary ammonium groups $X_2 = SO_4^{2-}$ or CO_3^{2-}) at pH 1.8 (acidic) and 10.5 (alkaline) media, respectively, and in low chloride concentration (not exceeding 2 g/l) [30].

In the present case, the concentration of chloride is higher than 10 g/l, which is 1,200 times higher than that of U(VI). The highest priority of the D263B resin was to adsorb the chloride; therefore, this resin was not suitable for uranium recovery.

3. Results and discussion

3.1. Ore processing and WPS characterization

One kilogram of sample of Allouga mineralized rock, with particle size 5: 10 mm, was packed up to 90 cm in a PVC column (100.0 cm long and a diameter 8 cm) and washed several times by slowly percolating water from the top of the column, thus giving time for the water to come into contact with the particles and dissolve the chloride as much as possible. The most effective washing was the half of the first bed volume (related to weight) or, in other words, the first 500 ml was characterized by high chloride and uranium concentrations. Chemical analysis of WPS is given in Table 2; chloride was decreased by washing till it reached to 1.5:2 g/l, and so the rock sample was ready for chemical processing [32–35].

Table 2
Metal ions' concentration in WPS after washing of ore

Element	Conc. (ppm)	Element	Conc. (ppm)
Si^{4+}	44	Cu^{2+}	24
Al^{3+}	79	Ni^{2+}	18
Ti^{4+}	8	Ti^{4+}	5
Fe^{3+}	76	U^{6+}	10.87
Mn^{2+}	190	Co^{2+}	6
Ca^{2+}	466	P^{5+}	70
Mg^{2+}	205	CO_3^{2-}	6,022
Na^+	600	Cl^-	13,340
K^+	42	SO_4^{2-}	1,079
Zn^{2+}	70	TDS	17,850

A very large volume of WPS produced during the processing of Allouga mineralized rocks after each recharge contained 10–12 mg/l uranium and 12–15 g/l of chloride (Fig. 7(a)). This contaminated solution was gotten rid of by recharging (Fig. 7(b)), or by keeping in vats and tanks for future evaporation (Fig. 7(c) and (d)).

During the washing process, the concentration of some ions, such as U(VI), Mg(II), Ca(II), and Na(I), was decreased. This decrease may be due to the effect of chlorides, carbonates, and sulfates on the mineralized rocks. The contents of these ions are shown in Table 3, and the ore material was now suitable for acid or alkaline leaching.

3.2. Loading process

As mentioned above, a large volume of WPS produced during the processing of Allouga ore was waste and not useable with QA resins, and cause pollution or other costs problems for restoring. The loading capacity of synthesized resins (GPAm and GPHA) was carried out by batch and column techniques. In batch method, the effects of loading time and pH were discussed. The column used was 10 cm high and of 1.5 cm diameter, and was packed with 5 g resin of each type with flow rate of 0.1–0.15 ml/min. The pH of the feeding solution had two values, 1.7 (optimum pH for D263B resin) and 7.2 (original pH).

In batch technique, 5 g of the resin was shaken in 1,000 ml of liquor. The uptake was determined at different times by the following expression:

$$q = (C_o - C_e) \times V \times W^{-1} \quad (3)$$

where q is the amount of metal ion adsorbed onto the unit mass of the resin (mg/g); C_o and C_e are the metal ion concentrations in the initial state and after loading upon the studied resin for certain period of time, respectively (mg/l); V is the volume of the aqueous phase (l); and W is the weight of the copolymer used (g).

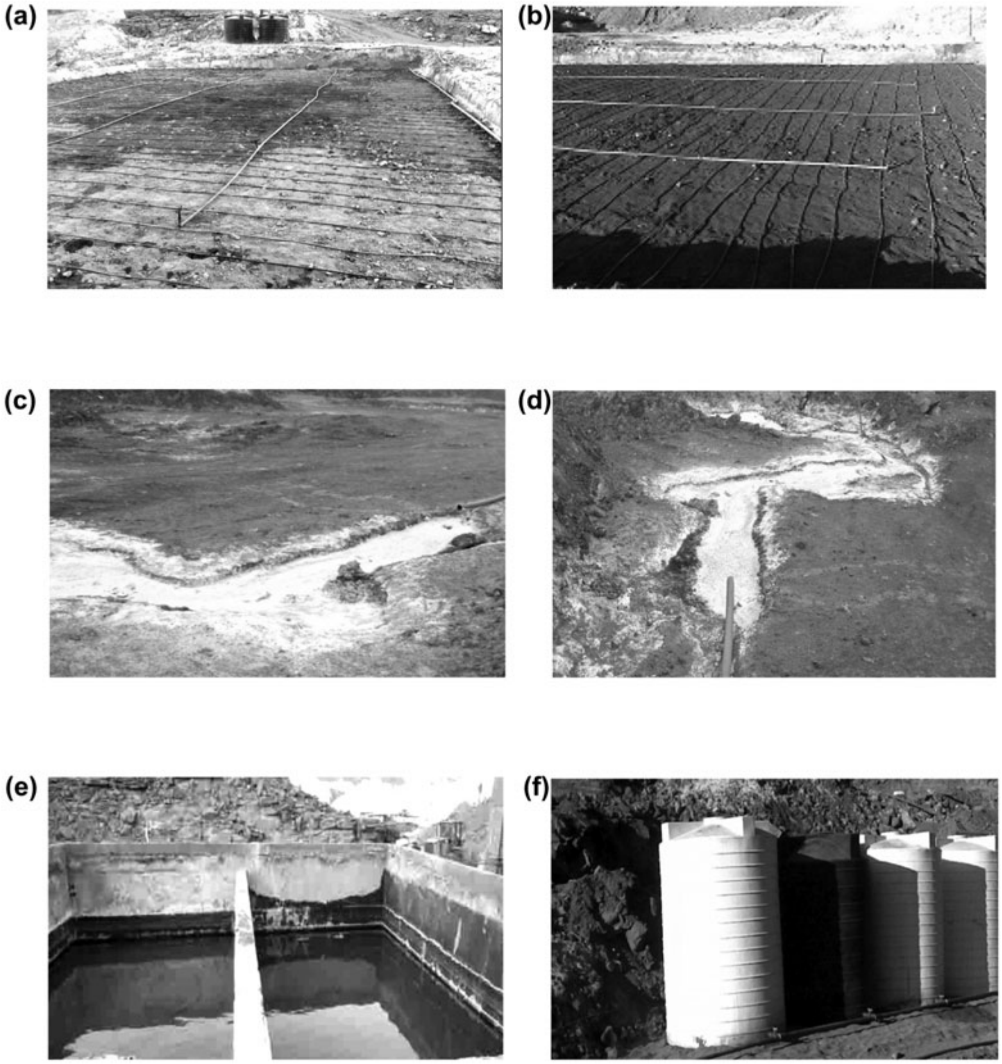

Fig. 7. Graphical photos: (a,b) preparation and washing of Allouga mineralization in vats; (c,d) removing of WPS; and (e,f) restoring of WPS in vats and tanks.

The most unique characteristic of GPAm and GPHA resins used was short loading time (that related to high swelling properties), high adsorptive capacity, low chloride affinity, large surface area, and the easy elution of adsorbed ions. The capacity of resins depended not only on the initial concentration of the metal ion but also on the pH of the solution. As the concentration of metal ions increased, loading capacity increased and time of saturation decreased [36,37].

Uptake capacity of D263B was 0.03 mg/g at pH 1.7, and 0.01 mg/g at pH 7.2; the concentration was extremely low by 2,000–3,000 times as compared by the optimum value of 60 mg/g. This could be directly attributed to the chloride content of 13.3 g/l.

3.2.1. U(VI) adsorption on GPAm resin

The loading capacity was increased by 3.6 times by increasing the pH of the solution from 1.7 to 7.2. The uptake on the column (40 mg/g) was slightly higher than that on batch method (39.5 mg/g) at pH 7.2 and it was 11 mg/g for both techniques at pH 1.7 (Fig. 8). Due to the basicity (negatively charges) of the amidoxime groups, chloride adsorption was decreased by nearly 5 times with respect to the D263B resin at the same pHs.

3.2.2. U(VI) adsorption on GPHA resin

The uptake of uranium on GPHA resin is shown in Fig. 8. The maximum loading capacities, at pH 7.2,

Table 3
Chemical analysis of ore residue after washing with freshwater

Element oxide	%	Element oxide	%
SiO_2	28.185	TiO_2	0.202
Al_2O_3	15.29	NiO	0.836
Fe_2O_3	9.695	U_2O_3	0.0289
MnO_2	1.3	RE_2O_3	0.01
CaO	13.672	P_2O_5	1.03
MgO	2.047	CO_2	3.4
Na_2O	1.58	Cl^-	2.1
K_2O	0.818	SO_4^{2-}	2.49
ZnO	2.11	LOI	14.9
CuO	0.22		

for both batch and column methods were 42 and 41.9 mg/g, respectively. At pH 1.7, it was 11.2 and 10.8 mg/g, respectively. Indeed, the low loading capacity at pH 1.7 was due to the protonation of amine groups making the pair of electrons unavailable for bonding with uranyl ions, thus decreasing the loading capacity as in *reaction 1*.

$$-NH_2 + H^+X \longrightarrow -NH_3^+X^- \qquad 1$$

The uptake of chloride on GPHA was lower than that on GPAm and QA resins at different pHs. This was due to the negatively charged amine group which repulsed the chloride ions.

3.3. Elution and precipitation

After loading, the resin was washed with a bed volume of distilled water followed by elution with 125 ml of 0.5 M HCl for 15 min in batch method or with a flow rate of 0.4 ml/min in column method. The uranium concentration in eluates from GPAm at pH 1.7 was 396 ppm in both batch and column technique, whereas at the same pH, the concentration yielded from GPHA resin was 369.9 and 460.8, respectively. At pH 7.2 in GPAm, the uranium concentration was 1,358.8 and 1,408, respectively, in which for GPHA it was 1,428 and 1,441, respectively as shown in Table 4.

The precipitation step was carried out at pH 8 using 10% NaOH solution [31]. The eluate from QA

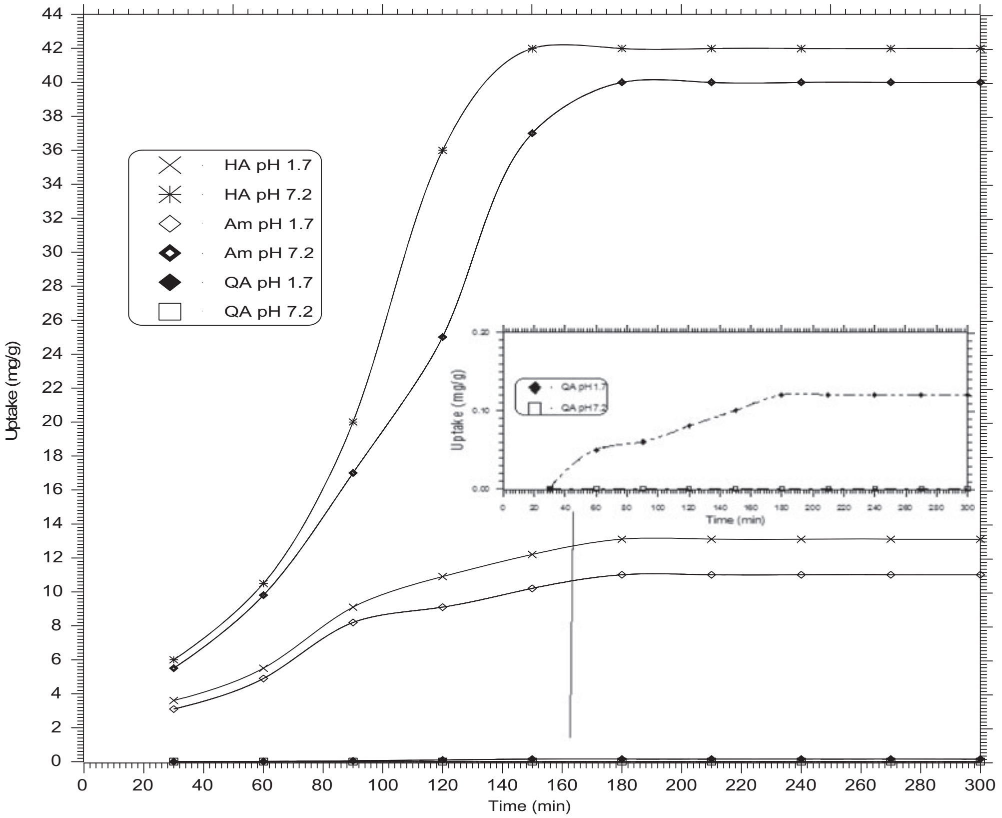

Fig. 8. Adsorption capacity of synthesized and D263B resins toward uranyl ions in batch techniques. *Conditions:* Initial uranyl ions concentration 10.87 mg/l, chloride concentration 13.34 g/l, and adsorption pHs, 1.7 and 7.2.

Table 4
Maximum loading capacity of resins (mg/g) and uranium concentration of eluates (ppm) and in precipitates (%)

Resin type	pH	Process used	U Loading cap. (mg/g)	Conc. of eluate (ppm)	Elution eff. %	U conc. in precipitate %
D263B	1.7	Column	0.1	3.9	97.5	–
	1.7	Batch	0.1	3.8	95	–
	7.2	Column	0.3	10.5	88	–
	7.2	Batch	0.3	10.7	89	–
GPAm	1.7	Column	11.0	396	93	22
	1.7	Batch	11.0	396	93	21
	7.2	Column	40.0	1,408	88	37
	7.2	Batch	39.5	1358.8	86	35
GPHA	1.7	Column	12.8	460.8	90	21
	1.7	Batch	13.2	369.9	89	21
	7.2	Column	41.9	1,441	86	40
	7.2	Batch	42.0	1,428	85	37

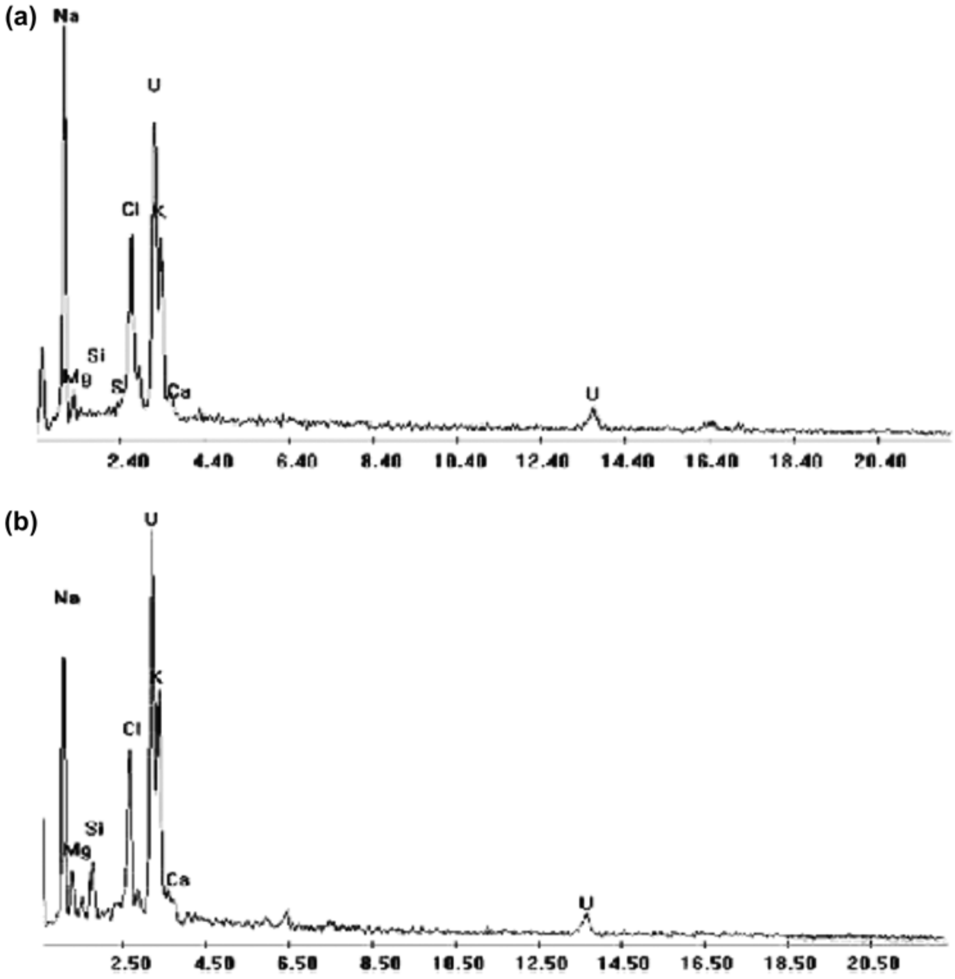

Fig. 9. EDX analysis of precipitate: (a) From GPAm resin and (b) from GPHA resin. *Conditions:* pH 8.0, precipitating reagent 10.0% NaOH.

resin was too dilute to be precipitated. The EDX analysis of the precipitates from GPAm and GPHA resins are represented in Fig. 9(a) and (b), respectively.

The concentration of effluents after loading from QA, GPAm, and GPHA resins were 10.85 ppm, 0.05 ppm, and 0.07 ppm with recovery percents of 0.26, 99.5, and 99.3%, respectively.

4. Conclusions

Allouga mineralizations are characterized by high uranium and chloride contents, causing major problems during the recovery of uranium; therefore, a systematic work in uranium recovery includes washing the ores with water in order to make it suitable for chemical processing. Moreover, the chloride competes with not only the adsorption of uranyl ions on QA resins (the most suitable tool for uranium recovery in industrial production scale) but also elute of adsorbed ions. For that reason, in this study, most attention was paid to the preparation of GPAm and GPHA resins, which have high affinity for uranium ions and low affinity for chloride ions due to the nature of the primary amine and hydroxyl groups. Also, these two resins, characteristically, have high swelling properties and high U(VI) uptake at various pH levels as compared to QA resins. It was found that slightly acidic pH was a good medium for uranium adsorption on synthetic resins and vice versa for QA resins. In the final analysis, the effluent solutions were made suitably environment-friendly. But the QA resin, due to its high chloride content, was not suitable for the removal of uranium. The final concentration of effluents after loading from GPAm, GPHA, and QA resins were 0.05 ppm, 0.07 ppm, and 10.85 ppm with recovery percents of 99.5, 99.3, and 0.26%, respectively.

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