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Solid phase extraction of uranium removal from underground water, Wadi Naseib, Southwestern Sinai, Egypt

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ABSTRACT

Water from well in Wadi Naseib is contaminated with uranium (4 mg/l). The maximum uranium concentration in drinking water is 0.009 mg/l as assigned by World Health Organization in 1998, thus this water increases drinking water limit nearly 500 times. It is characterized by high concentrations of carbonaceous matters and heavy metal ions reflecting the geochemical characterization of the surrounding rocks of black shale and dolomite. Removal of uranium was performed using commercial and synthetic adsorbents with respect to its effect on human health. Resins used were DOWEX 21K 16/20 type (commercial resin) and polyacrylamidoxime acrylic acid divinylbenzene PAm/AA (synthetic resin). DOWEX 21K 16/20 type is suited for mineral processing and groundwater remediation applications. PAm/AA has high capacity and selectivity for uranium and recovered it from low grade solutions. It was safe and suited for groundwater treatment.

Keywords: Groundwater treatment; Uranium contamination; Wadi Naseib; Organic sorbents

1. Introduction

Adsorption is an important technique in separation and purification processes which is used in water and wastewater industry to remove of color, odor, organic matter, and heavy metal ions pollution [1,2]. Although a variety of methods such as precipitation, solvent extraction, electrolysis, and ion exchange can be used to remove dissolved metal ions from aqueous samples, most of them have disadvantages of non-economic, poor removal efficiency, high cost, generation of pollution, and ineffectiveness for low metal concentrations [3]. Many types of adsorbent materials are known specially nitrogen and oxygen containing groups as amine, carboxylic and amidoxime groups, which have low cost and classified as high adsorptive materials category [4,5]. Resins containing amide, amine, carboxylic acid, sulfonic acid, and ammonium groups not only possess good hydrophilic properties, but also can bind to metal ions from aqueous solutions [6]. Adsorption of uranium onto various solids is important from purification, environmental, and radioactive waste disposal points of view [7]. The maximum uranium concentration in drinking water is reported as less than 0.009 mg/l [7]. For this purpose, new sorbent materials such as polymeric resins, activated carbon, naphthalene, and silica gel have been developed for more effective extraction [8–12]. However, some of the sorbents suffer from a number

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of drawbacks such as long pre-concentration time, low mechanical stability of the sorbent, slow kinetics, irreversible adsorption of target, and swelling [13–19]. Some of the reported chemically modified sorbents are those containing ligands with oxygen and nitrogen donors involved in U(VI) chelation forming for selective extraction of U(VI) from various matrix components [20–22].

The selective adsorption of metal ions depends on a small difference in the stability constant of the complex between a polymer legend and a uranyl ion.

The main target of this work is the extraction of uranium from contaminated underground water having high content of heavy metals and organic matter.

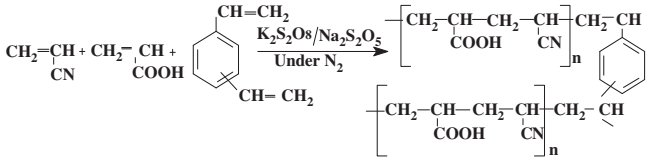
2. Experimental

2.1. Materials

Dowex 21 K and Divinylbenzene (DVB, 55%) were obtained from Sigma-Aldrich, England. Acrylonitrile, acrylic acid was obtained from Fluka, Japan. Potassium persulfate and sodium metabisulfite were obtained from Adwec Co, Egypt. All other chemicals were Prolabo products and were used as received.

2.2. Synthesis of Polyacrylonitrile Acrylic Acid resin

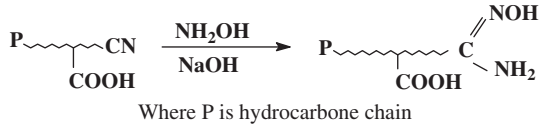
Polymerization process was performed by reaction of acrylonitrile, acrylic acid and divinyl benzene with molar ratio 50:50 and 10%. Polymerization occurred at 40 °C under nitrogen. Mixture of Potassium persulfate and sodium metabisulfite (redox initiator) was added to Poly vinyl alcohol (1%) and then monomers were added in the above mixture in 25–30 min followed by 60 min good agitation at 60°C. Polymer was isolated by filtration, washed with water and methanol and dried under vacuum at 50°C (Scheme 1) [23,24].



Scheme 1. Synthesis of PAN/AA copolymer.

2.3. Synthesis of Polyacrylamidoxime Acrylic Acid resin

To 30 g of Polyacrylonitrile Acrylic Acid (PAN/AA) resin, 21.1 g of hydroxylamine hydrochloride in 290 ml methanolic solution (methanol:water is 5:1, respectively) at pH 10 was added. The reaction mixture refluxed at 70°C for two hours. Resin was separated by filtration and washed several times by methanolic solution and then with 0.1 M HCl solution for at least 5 min. Finally, it was filtered off, washed with methanolic solution, and dried at 50 °C to constant weight (Scheme 2) [25].



Scheme 2. Synthesis of PAm/AA copolymer.

2.4. Analytical techniques

Resin contacted with water sample on column and batch method at natural pH 6.8. Uranium was measured in water samples and in effluents using ICP-MS. After precipitation, it was analyzed using spectrophotometer by arsenazo III.

2.5. Loading process

Loading was performed in column and batch techniques. Column has specification of 10 cm length and 1.5 cm diameter; it backed with 5 g of each dried resin. Flow rate was the main factor affecting on adsorption, the optimum flow rate was 0.03 ml/min. In batch method, effect of time was the main factor that is responsible in adsorption capacity and it was increased by time. The uptake was calculated by using the following expression:

$$q = (C_0 - C_e) \times V \times W^{-1}$$

where q is the amount of UO_2^{2+} adsorbed onto unit mass of the copolymer (mg/g), C_0 and C_e are the concentrations of the uranium in the initial solution and in the aqueous phase after treatment for certain period of time, respectively (mg/l), V is the volume of the aqueous phase (l), and W is the mass of the copolymer used (g).

3. Results and discussion

3.1. Characterization of DOWEX 21K 16/20 type

It is a strong base anion resin, insoluble in most of common solvents, highly ionized, stable till temperature up to 60°C, usable over highly pH range, stable in

absence of strong reducing or oxidizing agents, have fast elution rate, excellent kinetics, excellent regeneration efficiency, and outstanding physical stability. It is suited for mineral processing and groundwater remediation applications. It has resistance to organics and fast equilibrium rates. It is suited for fluidized-bed and resin-in-pulp applications.

3.2. Characterization of synthetic resin

The main characteristic features of Polyacrylamidoxime Acrylic Acid (PAm/AA) resin with this molar ratio and cross-linking percent were discussed in previous work and summarized in below items [25].

Completely, conversion of nitrile groups to amidoxime has high chemical and mechanical stability that reflected in the soluble fraction percent, elution and regeneration process. It has high porosity that allows ions to diffuse inside pore in short period of time and has high affinity for recovering ions from low concentrate solutions. The porosity confirms the three-dimensional structure of the hydrogel, high swelling properties, and consequently highly uptake of metal ions in short period of time [24,25]. Types of adsorption of uranyl ions on quaternary amine and amidoxime carboxylic groups are shown in Fig. 1 [26].

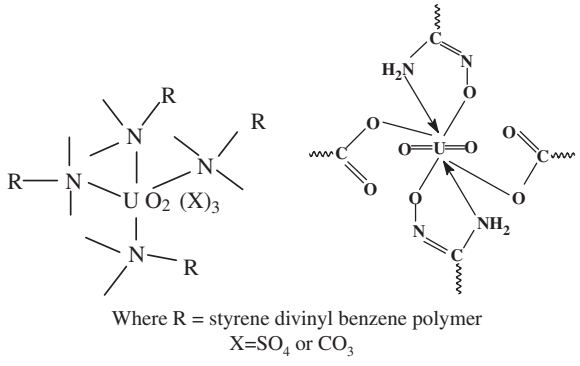


Fig. 1. Type of bonding of uranyl complexes with acrylamidoxime acrylic acid and quaternary amine resins.

3.3. Water characterization

The water sample was taken from newly dug well in Wadi Naseib by Drinking Water and Wastewater Company, it is far from sea water by nearly 40 km, the position of Wadi Naseib in geological map [27] was mentioned in Fig. 2. The water surface in this well is about 2 m from the ground surface. This is due to the presence of a basaltic dyke extruding the sandstone aquifer of Adediya Formation which led to the rise of water against the dyke to be near from the ground surface. Withdrawal of 10 m^3 of water can be recharged within 24 h. The surrounding rocks are mainly of black shale and dolomites with ferruginous sandstone. This formation is consists of lithologies with high uranium organic matter contents [28].

The chemistry of water is shown in Table 1. It has high concentrations of anions as SO_4^{2-}, Cl^-, and CO_3^{2-}; cations as Mg(II), Na(I), Ca(II), and Al(III); and heavy metal ions as Mn(II), Zn(II), Ni(II), and Cu(II). Uranium is the most important element in this study which is present in concentration of 4 mg/l. This high concentration is due to its complexation with carbonate [24].

Table 1
Analysis of elements in studied water well

Element	$mg\,l^{-1}$	Element	$mg\,l^{-1}$	Element	$mg\,l^{-1}$
SiO_3^{2-}	90	Ca(II)	762	Ni(II)	170
Al(III)	260	Mg(II)	214	U(VI)	4.0
TiO_3^{2+}	10	Na(I)	1,080	CO_3^{2-}	2,028
Fe(III)	70	K(I)	102	Cl^-	2486
Mn(II)	220	Zn(II)	190	SO_4^{2-}	928
		Cu(II)	98	TDS	6,860

Adsorption of metal ions on resins is described as liquid–solid phase reaction which includes the diffusion of the ions from solution to resin surfaces and pores. The most effective parameters on adsorption are initial concentration of ions, loading time, pH of feeding solution, and competitive ions of resins, especially chloride and nitrate.

As time of loading increased, efficiency of adsorption increased. The stability of resins was determined not only by remaining loaded with metal ions as long time as possible but also by reaching to maximum saturation in short time [29,30].

As pH increased, adsorptive capacity of resins increased, due to increasing availability of lone pair of electrons on nitrogen and oxygen; the effect of loading time and pH was discussed later [24,30]. It was found that as concentration of metal ions increased, adsorption efficiency increased and saturation time decreased [31].

Water has high concentration of organic matter (carbonaceous) and some elements as Ca(II), Al(II), Zn (II), Fe(II), Cu(II), and U(VI). This is reflected in geochemistry of the surrounding rocks which are Siltstone (silica, calcite, and iron oxides are the most common minerals), Dolostone (the main constituent is the mineral dolomite $(CaMg)(CO_3)_2$), and Black shale (Organic matter, pyrite, and minerals containing carbonate mineral, quartz, calcite, iron oxide minerals,

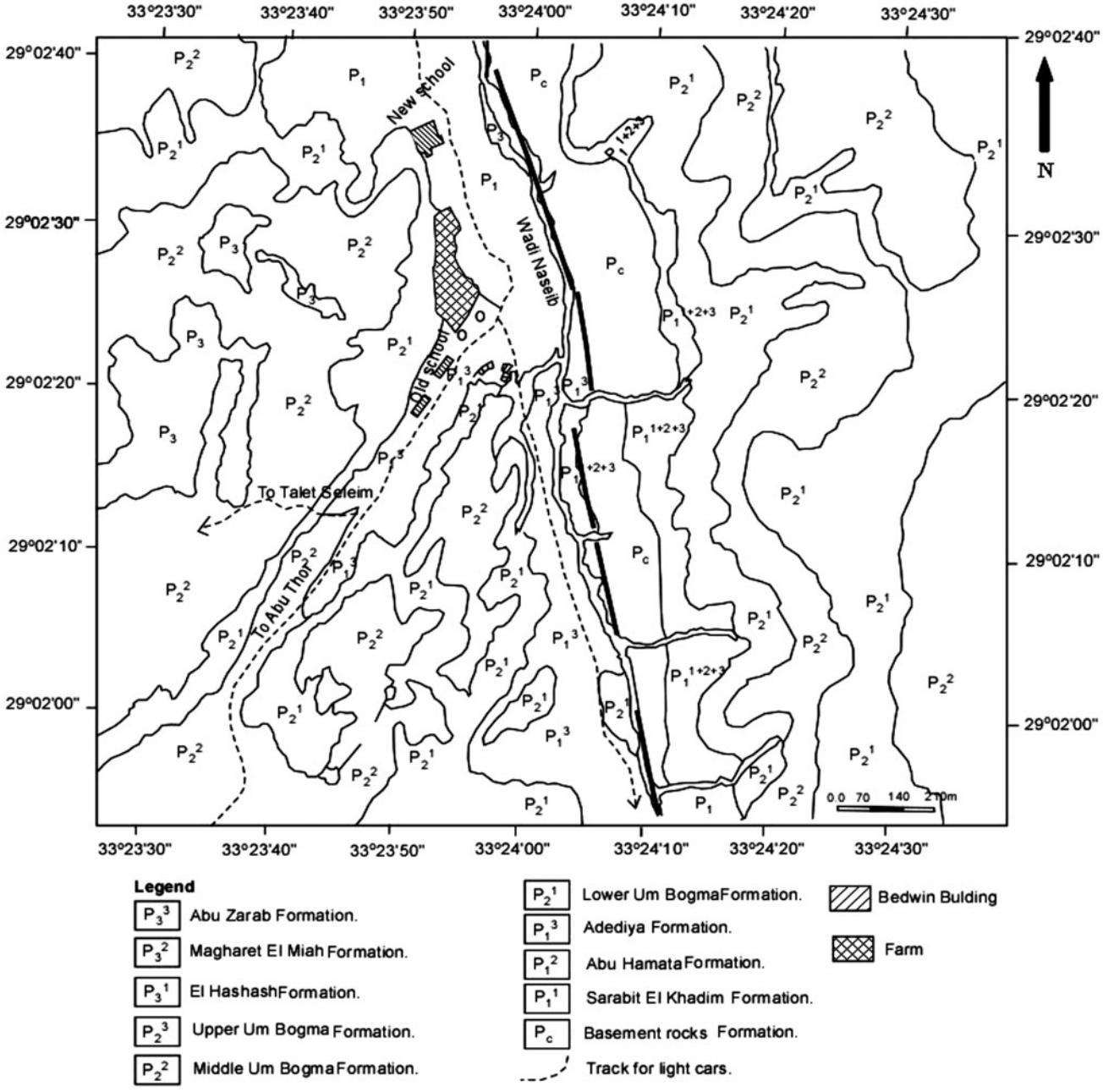


Fig. 2. Geologic map of the studied area with location of dug water well (After Elassy 2000 [27]).

and sulfide minerals) and this is shown in the analysis of surround rocks (Table 2).

3.4. Loading process

3.4.1. Column process technique

On column with specification of 10 cm length and 2 cm diameter, 5 g of dried resins were packed. Different flow rates were performed (from 0.01 to 0.06 ml/min) and the maximum uptake of each resin is listed in Table 3.

The optimum flow rate was 0.03 ml/min that gives higher uptake capacity than others, low flow rates than 0.03 cause adsorption of other ions as chloride and carbonate as well as organic matters, whereas higher flow rates cause insufficient time for uranium adsorption. Uranium concentrations were determined in each 100 ml of effluent and the relation between C/C_0 and volume taken are represented in Fig. 3, in

Table 2
Analysis of metal oxide in the surrounded rocks of water well

Metal oxide%	Dolostone	Siltstone	Black shale
SiO_2	22.49	48.32	32.80
Al_2O_3	10.50	10.60	11.76
TiO_2	0.11	0.83	0.68
Fe_2O_3	6.89	20.42	20.79
MnO_2	0.79	0.15	0.09
CaO	20.34	4.14	1.82
MgO	11.56	2.91	1.27
Na_2O	1.72	1.70	1.50
K_2O	0.62	0.49	2.38
ZnO	1.1	0.9	2.1
NiO	1.4	1.1	0.9
CuO	0.09	0.03	0.1
U_2O_3	0.36	0.3	0.4
P_2O_5	0.05	0.16	3.59
Cl^-	2.4	1.3	1.6
SO_3^{2-}	0.9	0.77	2.10
LOS	18.7	6.7	17.8

LOI = loss on ignition.

Table 3
Effect of flow rate (ml/min) on uranium adsorption (mg g^{-1}) of Dowex 21 K 16/20 type and PAm/AA resins

Flow rate (ml/min)	Adsorption capacity (mg/g)	
	Dowex 21 K	PAm/AA
0.01	0.83	3.8
0.02	0.86	3.9
0.03	0.92	3.96
0.04	0.89	3.09
0.05	0.81	3.0
0.06	0.6	3.0

which C is the effluent concentration and C_0 is the initial concentration of uranium in water. $C = C_0$ or $(C/C_0 = 1)$ when outlet equal to inlet or on other words when resin being saturated, volume of feeding solution as in DOWEX 21 K 16/20 type lower than in PAm/AA and this would mean low performance of DOWEX, also high uptake of PAm/AA.

Low capacity of DOWEX resin (0.92 mg/g) due to relatively high concentration of chloride and carbonate that make competition with U(VI) causes a definite decrease in loading capacity, also low concentration of it was the main factors on decreasing of loading capacity. High capacity of PAm/AA (3.96 mg/g) than DOWEX resin is due to highly swelling properties and large surface area that is recognized as the main

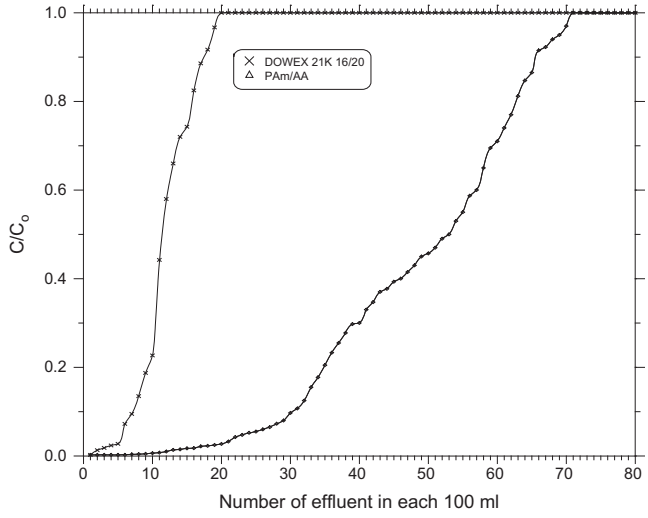


Fig. 3. Loading capacity of resins on column process of each 100 ml of effluent.

factors on adsorption, especially on low metal ions concentration, and also low affinity of amidoxime and carboxylic groups toward chloride and carbonate ions increasing capacity of uranium loading.

3.4.2. Batch method technique

In 10 liter of water sample, 10 g of each resin was agitated. This experiment was performed at two different conditions. First batch experiment (first exp.), in which loading process takes place for 50 h and loading efficiency was determined. Second batch experiment (second exp.) was performed on synthetic resin in which loading was carried out in five interval stages each with 10 h, and the capacity was determined after loading of each stage.

Time and pH are the main factors affecting on adsorption. The capacity of DOWEX 21 K 16/20 type resins was very low compared to PAm/AA, it was

Table 4
Second experiment of uranium loading on PAm/AA resin

Contact time (hour)	Initial conc. (mg/l)	Conc. In effluent (mg/l)	U loaded on resin (mg/g)	Uptake efficiency (%)
1st 10 h	4	3.62	0.38	9.5%
2nd 10 h	3.62	2.9	0.72	19.89%
3rd 10 h	2.9	0.67	2.23	76.9%
4th 10 h	0.67	0.08	0.59	88%
5th 10 h	0.08	0.01	0.07	87.5%

Table 5
Analysis results of water sample after loading on resin

Metal ions	Column tech.	Batch method		Metal ions	Column tech.	Batch method	
	Conc. mg/l	First exp.* Conc. (mg/l)	Sec. exp.** Conc. (mg/l)		Conc. mg/l	First exp.* Conc. (mg/l)	Sec. exp.** Conc. (mg/l)
U(VI)	0.039	0.028	0.01	Fe(III)	40	54	10
SO_4^{2-}	240	394	202	K(I)	86	61	16.1
Cl^-	1,001	1,000	902	Mn(II)	122	124	93
CO_3^{2-}	370	1,005	500.4	Zn(II)	23	19	12.4
Mg(II)	16	12	9.9	Ni(II)	29	21.2	15.7
Na(I)	755	600.1	341.1	Cu(II)	12	11.04	7.09
Ca(II)	277	123.4	102.2	SiO_3^{2-}	19	12.1	10.3
Al(III)	176	151.6	113	TDS	1880	1,654	2,320

* First exp., the uptake measured after 50 h.
** Sec. exp., the uptake measured on five intervals each one was 10 h.

0.82 and 3.97 mg/g of each resin, respectively. The final concentration of uranyl ions in water sample was 3.18 and 0.028 mg/l with recovery 20.5 and 99.3%, respectively. In second experiment (Table 4), the most adsorbed in first two steps (20 h) were organic matter (carbonaceous) and heavy metal ions as Zn(II), Ni(II), Cu(II), Fe(III), and TiO_3^{2+}. This was expected due to high concentration of this ions compared to U(VI) and high ability of amidoxime groups to it [32].

As time increase, efficiency of uranium uptake increased, that reached to 9.5% and 19.89% after first and second 10 h, respectively. Concentration of uranium after removing these ions was 2.9 mg l^{-1}, which is recognized as higher in concentration by 360 times with respect normal drinking water.

Concentration of uranium in water became 0.01 mg l^{-1} that will be suitable for human and animal uses. The final recovery of this experiment was 99.75% for 50 h (divided to five intervals). Concentration of Ions in water after loading on PAm/AA resin in each experiment is exhibited in Table 5. It was noticed that the capacity of removing of metal ions in second experiment is higher than first one.

Table 6
Elemental analysis % of result precipitates after elution from resin

Metal ions	Column experiment	*First batch experiment (50 h)	**Second batch experiment			
			First precipitate after 1st 10 h	Second precipitate after 2nd 10 h	Third precipitate after 3rd 10 h	Fourth precipitate after 4th 10 h
C	13.7	10.00	48.2	–	–	–
Na^+	3.6	1.14	9.39	5.09	–	–
Mg^{2+}	4.1	2.7	17.52	13.12	–	–
Al^{3+}	6.9	3.21	7.0	3.01	–	–
S^{2-}	3.89	2.16	3.09	6.65	0.89	–
Cl^-	5.5	5.06	7.19	2.44	–	3.5
Ca^{2+}	6.8	7.15	10.33	14.83	0.99	8.67
Mn^{4+}	4.6	5.01	15.6	13.23	–	4.26
Zn^{2+}	4.9	2.87	10.76	12.98	–	2.49
U^{6+}	19.9	40.19	9.4	20.69	98.12	71.1
Ni^{2+}	3.1	2.5	2.74	2.66	–	–
Cu^{2+}	2.9	2.7	1.98	1.98	–	–
Si^{4+}	7.8	6.07	5.00	3.32	–	1.14
Fe^{3+}	9.9	8.21	–	–	–	1.14
K^+	2.4	1.03	–	–	–	7.7

∗ First exp., the uptake measured after 50 h.
∗∗ Second exp., the uptake measured on five intervals each one was 10 h.

Table 7
Correlation values of adsorption capacities of U(VI) by different adsorbents

Chelating resins	Adsorption capacity mg/g	Analyzed sample	Reference
Support: Amberlite XAD-4 O-vanillin semicarbazone	2.38	Simulated river water	[10]
Piroxicam	1.9	River and marine	[13]
Support: Molecularly imprinted polymers 5,7-dichloroquinoline-8-ol	2.618	Sea water and sediment	[14]
Trifluoroethylxanthate Naphtalene	1.666	Sea water	[14]
Support: Octadecyl-Tri-n-octyl phosphineoxide silica membrane disc	3.522	Natural water	[15]
Tri-n-octyl–phosphineoxide ctadecyl-silica membrane disk	3.046	River and well water	[15]
Support: Naphthalene/ Benzophenone 1-(2-Pyridylazo)2-naphthol	2.14	River and marine	[19]
Support: Amberlite XAD-2 Pyrogallol	3.095	River and well water	[34]
Carboxylate functionalized graft copolymer PGTDC-COOH	2.99	Natural water	[35]
Amberlite XAD-4-OVSC	2.38	Well water	[36]
Grafted amidoxime resin	0.5	Sea water	[37]
1-(2-Pyridylazo) naphthol	2.55	Sediment and soil	[37]
PAm/AA	3.97	Well water	Present work
DOWEX 21 K	0.82	Well water	Present work

3.5. Elution of Uranyl(VI) ions

Elution was carried out by 4 N HCl, each 1 g resin eluted by 5 ml eluent. This highly concentrate acid used to emphasis that resin's structure not affected by highly acid concentration (changing of its chemical structure) that 0.5 M HNO_3 can elute higher than 90% of adsorbed ions. Results of elution in column gives 25 ml eluate with concentration of 753.9 mg l^{-1} and 180.3 mg/l for PAm/AA and DOWEX resins, respectively, with elution efficiency 95.2% and 98%, respectively. On batch method, the concentration in 50 ml eluate was 765.3 and 161.7 mg/l with efficiency 95.9 and 98.9%, respectively. Concentration in each step of second batch experiment was 74.8, 141.5, 437.5, 116.8, and 13.8 mg/l with efficiency 98.5, 98.3, 98.1, 99, and 99.1%, respectively. The picture of resulted water (Table 5) in the second experiment can be accepted for different purposes.

3.6. Precipitation of dissolved uranyl ions

Uranyl(VI) ions were precipitated by NaOH at pH 7. Yellow cake precipitation was filtered off and then dried. Analysis of precipitate is represented in Table 6, which indicated that each stage has different uranium content than other depending on process used. Fifth step in second batch experiment was too dilute to be precipitated.

Uptake of uranyl ions on PAm/AA were relatively high compared with other adsorbents [33–37] as in Table 7, that each 1 g PAm/AA resin loaded by 3.98–3.95 mg uranium.

4. Conclusion

The main source of water in Wadi Nassib is ground water. It is contaminated by 4 mg/l uranium which is recognized as very high and dangerous for environment and human health. Synthesized organic adsorbents bearing amidoxime and carboxylic groups show high affinity for uranium removing. PAm/AA resin has a higher loading capacity than commercial resins used in uranium removal from ground water than DOWEX 21 K 16/20 type. It has a stable structure and high mechanical stability which resist back feeding by increasing time of loading. Loading was performed in two processes, column and batch techniques, each one showing high removal efficiency for uranium. The final concentration in ground water was suitable for human use. High purity of U(VI) precipitate in the presence of high content of heavy metals and organic matters performed by two adsorption steps was used before.

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