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SOLID-LIQUID PHASE EXTRACTION OF THORIUM (IV) FROM NITRATE LEACH LIQUORS USING AMINE FUNCTIONALIZED GLYCIDYL METHACRYLATE RESINS

MAHMOUD O. ABD EL-MAGIED; EL-SAYED A. MANAA; MOHAMED N. KOURAIM; MOHAMED A. YOUSSEF and EKRAMY M. EL-DESOUKY

Nuclear Materials Authority, Cairo, Egypt

ABSTRACT

Thorium adsorption from low concentration nitrate leach liquors was studied using glycidyl methacrylate/divinyl benzene/amine polymers. RI and RII resins were prepared by the reaction of GMA/DVB copolymers with ethylenediamine and diethylenediamine, respectively. The thermal stability of the resins was studied thermogravimetrically. The prepared resins were applied for the adsorption of thorium from synthetic and real samples. The results indicated that; the adsorption of Th(IV) increased as the solution pH increased and the maximum metal adsorption was 50 and 64 mg/g for RI and RII, respectively. Thorium (IV) adsorbed per mass unit of RI and RII resins increased from 50 to 64 and from 66 to 88 mg/g by increasing the temperature from 293° to 323° K respectively. The obtained results indicated the applicability of RI and RII for Th(IV) adsorption. Finally, RI and RII resins were used for the separation of Th(IV) from three granite samples leach liquors.

INTRODUCTION

Thorium species are distributed in nature in many minerals such as thorite [ThSiO₄], thorianite [ThO₂], uranothorite [(Th, U).SiO₄] beside monazite [Ce, La, Y, and Th) PO₄] which was considered as the most important thorium source (Eisenbud and Gesell; 1997). Thorium is considered as an important metal which required for several applications as catalysts, high-strength, high-temperature alloys and in medicine beside it can be used in the future nuclear fuel in nuclear applications (Habashi, 1997; Borai and Mady; 2002). The previous applications produce various toxic and non-degradability thorium wastes

causing many environmental problems. The chemical toxicities of these wastes depend on thorium solubility especially in the biological media (Friedlander et al., 1981; Keogh; 2006). Thorium ions enter the human body by inhalation, through the skin or mucous membranes besides it can be absorbed through the lungs. The extensive hazard applications of the development of reliable methods for thorium leaching using different techniques from the environmental and geological wastes is a strategic goal in economic and environmental views (Vertes et al., 2010; Riazi et al., 2014; Zhou et al., 2016; Hu et al., 2016; Jing et al., 2016; Kirkan and Aycik, 2016). Liquid-liquid extraction has remained the preferred tech-

due to its selectivity but has several problems mainly the organic wastes generation (Hen-nion; 1999). In contrast, the solid-phase extraction technique has turned out to be a more eminent and promising method, owing to its advantages for metals recovery because of its simplicity, reliability, high selectivity, high capacity, environmental safety, low cost, and low solvent consumption. Glycidyl methacrylate is an interesting monomer based on its own active ethoxy group (oxirane). Poly glycidyl methacrylate-based polymers were used in metals recovery (Donia et al.; 2009, Cannero et al.; 2009, Sadeek et al., 2014; Abd El-Magied, 2016). This work aims to study the adsorption of Th(IV) ions from aqueous solutions by modified glycidylmethacrylate resins. The kinetic and isotherm models were used to fit the adsorption process.

EXPERIMENTAL

Instrumentations, Chemicals and Analysis

The pH values were adjusted using pH-meter (Digimed DM-21, Japan). The flasks were shaken using ashaking water bath (Labortechnik mbH-1083, Germany). The thermal analysis measurements were performed using a simultaneous DTA-TGA thermal analyzer (Shimadzu DTG-60, Japan). Thorium(IV) concentrations in the aqueous solutions were estimated spectrophotometrically using Thoron 1 (UV/VIS spectrophotometer, Labomed, Inc. USA) method (Marczenko, 1986). All chemicals were of analytical grade. Glycidyl methacrylate, divinylbenzene, ethylenediamine, and diethylenetriamine are Sigma-Aldrich products.

Resins Preparation

The resins were prepared as presented by Sadeek et al. (2014) with some modification. Glycidyl methacrylate/divinyl benzene (GMA/DVB) copolymers were synthesized by suspension copolymerization of the glycidyl methacrylate monomer and divinyl

benzene as an across linker in the presence of a 2,2-azobisisobutyronitrile as initiator and polyvinyl alcohol as a suspension stabilizer. The functionalized RI and RII resins were prepared by adding the obtained GMA/DVB copolymers in N,N-dimethylformamide containing ethylene diamine and diethylene triamine (Fig. 1).

Adsorption Measurements

In order to study Th(IV) ions adsorption from aqueous solutions using the prepared RI and RII resins; some parameters including pH, solid/liquid phase ratio, contact time, initial Th(IV) concentration, etc. were studied and optimized. Thorium (IV) concentrations before and after adsorption processes were determined spectrophotometrically while the adsorption capacity (q , mg/g) was calculated, from initial and final ion concentrations (C_i and C_e , mg/L), volume V (L), and weight W (g), using Eq. 1 (Donia et al., 2009):

$$q = \frac{(C_i - C_e)}{W} V \quad q = \frac{(C_i - C_e)}{W} V \quad (1)$$

General Procedures

Generally, 50 mg of the RI or RII resins, as adsorbent, were added to a series of flasks

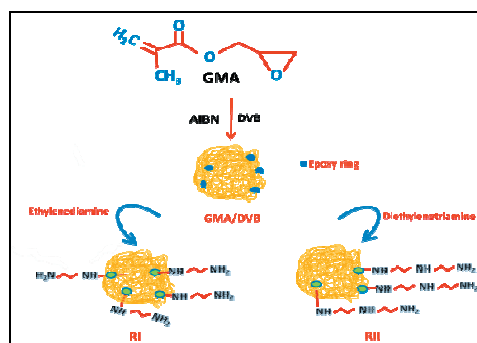


Fig. 1: Synthesis procedures of RI and RII resins

contains a 100 ml aqueous solution. The flasks were then shaken at 300 rpm for 15 min. After equilibrium, the resins were filtered and the residual Th(IV) ions concentration were determined to predict the adsorption capacities at the different experimental conditions. The experimental data were applied to different isotherm and kinetic models.

RESULTS AND DISCUSSION

Resins Specifications

FT-IR analysis

FTIR spectra of the prepared cross-linked GMA/DVB, RI and RII resins are present on Figures (2& 3). The obtained data indicated that the band at 3300-3700 cm^{-1} corresponds to the stretching vibration of the N-H and O-H groups. On the spectrum, there is a band at 2925 cm^{-1} for C-H stretch (alkyl). Bands at 1650-1750 cm^{-1} (C=O stretching) and at 1600 cm^{-1} (benzene groups) appear in the spectra. Bands at 1470 cm^{-1} attributed to scissoring of C-H. The IR region below 1500 cm^{-1} is known as the fingerprint area due to bending motion. The bands at 1200-970 cm^{-1} are related to the C-C and C-O stretching in-

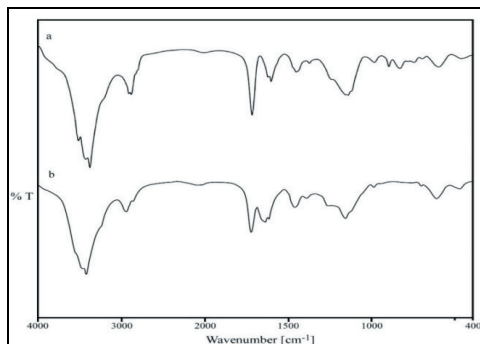


Fig. 2: FTIR spectrum of GMA/DVB (a) compared to RI resin (b)

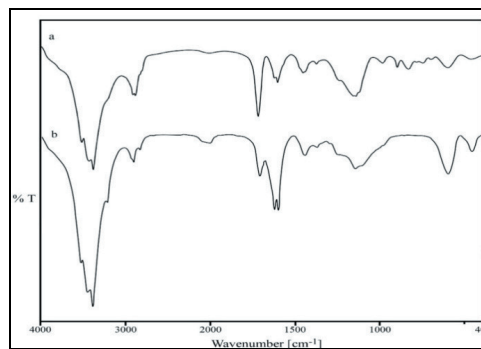


Fig. 3: FTIR spectrum of GMA/DVB (a) compared to RII (B) resin (b)

plane, where the bands at 950-910 cm^{-1} are related to the O-H bending of the bonds.

Thermal analysis

Thermogravimetric (TG/DTG) analysis was utilized to obtain quantitative information on weight losses as a function of time and temperature in conjunction with a mass spectrometry analysis (Salvio-Neto and Matos, 2011). The TGA curve refers to three stages of mass losses at temperature ranges 25-268.77, 268.77-408.14 and 408.14-650 $^{\circ}\text{C}$ (Figs. 4&5). These stages involved mass losses of 5.9, 31.08 and 26.86% for RI resin decomposition respectively. For RII resin decomposition, also there are three stages as temperature ranges of 25-268.77, 268.77-408.14 and 408.14-650 $^{\circ}\text{C}$ have mass losses of 10.28, 35.54 and 55.82%, respectively. From the obtained results, we noticed that; RI resin is more stable than RII. For RI resin, the endothermic peak at 206.63 $^{\circ}\text{C}$ is due to the melting, peak at 315.55 $^{\circ}\text{C}$ is attributed to the first decomposition corresponding to the first mass loss observed in TG and the sharp exothermic peaks at 454.26 $^{\circ}\text{C}$ is due to the complete decomposition of the compound. The DTA curve of RII shows that all peaks, at 126.13, 315.55 and 454.92 $^{\circ}\text{C}$, are endothermic peaks.

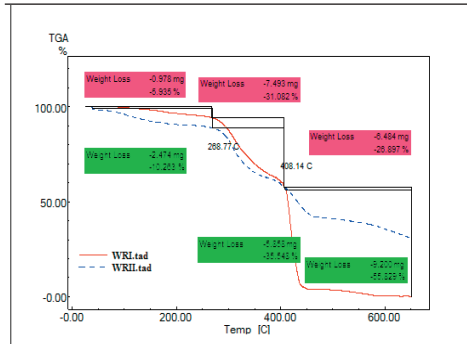


Fig. 4: TG-thermo grams of RI and RII resins

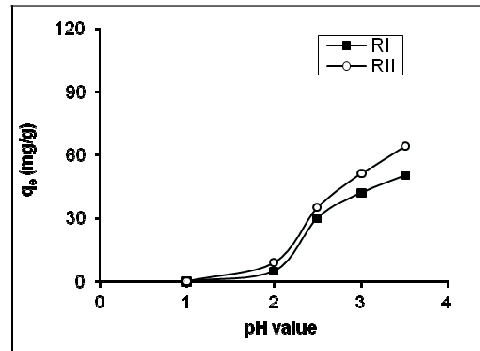


Fig. 6: Effect of pH on the adsorption of Th(IV) ions by RI and RII resins

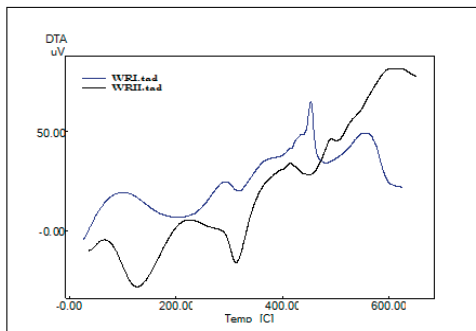


Fig. 5: TGA/DTG curves of RI and RII resins

Adsorption and Desorption Factors

Effect of pH

Generally, the aqueous solution pH is a very important parameter for metals adsorption processes using resins. Studies of Th(IV) adsorption behavior on RI and RII resins were performed at various pH values ranged from 1 to 4 using dilute HNO_3 and NaOH solutions. Fifty mg of the resin was added to with 100 ml aqueous solution, containing 100 mg/L Th(IV) ions. The obtained results on Figure (6) indicated that; the adsorption of Th(IV) using RI and RII increased with increasing the medium pH value until pH 3.5. At a pH of 3.5; maximum adsorption capacity of RI and RII were found to be 50 and 64 mg/g respectively.

Very low thorium ions adsorption efficiency obtained at $\text{pH} < 1.5$; which might due to that, at excess hydrogen (high acidic medium) ions a competition between H^+ and Th(IV) ions on the binding sites occurred which led to decreasing Th(IV) adsorption (Sadeek et al., 2014). On the other hand, raising the solution pH value than 3.5; led to Th(IV) precipitation and hydrolysis according to Eq. 2. The monomeric hydrolyzed species, including $\text{Th}(\text{OH})_2^{2+}$ and $\text{Th}(\text{OH})_3^{3+}$ at $\text{pH} \geq 3.7$, begin to precipitate as $\text{Th}(\text{OH})_4$ (Cornelis et al., 2005; Cotton, 2006). The optimum aqueous pH values for maximum Th(IV) adsorption using RI and RII resins are 3.5.



Effect of contact time

The effect of time of Th(IV) ions adsorption on 50 mg of RI and RII resins within series aqueous solutions containing 100 ml containing 100 mg/l Th(IV) ions (S/L of 1/2) at pH 3.5. The solutions were shaken for different periods ranged from 5 to 120 min. The obtained results on Figure (7); show that; a rapid adsorption rate was achieved within 5 min and the maximum adsorption equilibrium was achieved after 15 min for both RI and RII resins.

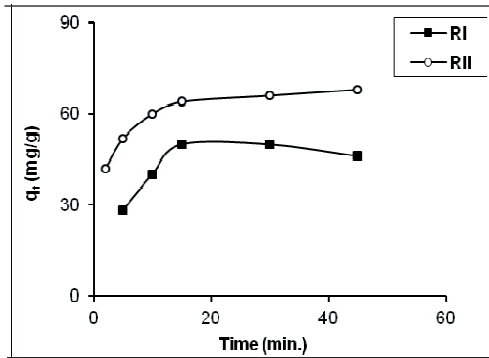


Fig. 7: Effect of contact time on the adsorption of Th(IV) ions by RI and RII resins

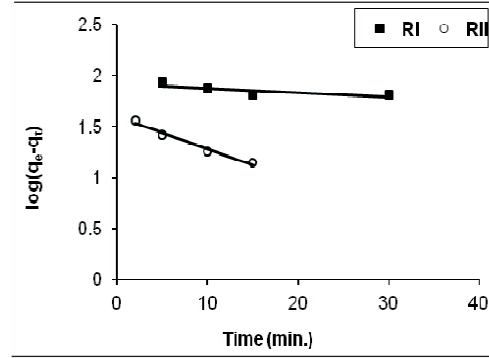


Fig. 8: Pseudo first order plots for adsorption of Th(IV) ions on RI and RII resins

Kinetic studies

Some kinetic performance measurements of RI and RII resins were studied in this work. The time-dependent data were treated according to the pseudo-first and pseudo-second-order kinetic models (Figs. 8&9). The kinetic parameters, adsorption capacity (q_e and q_t , mg/g) and rate constant (k_1 and k_2), were calculated according to Eq. (3, 4).

$$\log(q_e - q_t) = \log(q_{1st}) - \frac{k_1}{2.303} t \log(q_e - q_t) = \log(q_{1st}) - \frac{k_1}{2.303} t \quad (3)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_{2nd}^2} + \frac{1}{q_e} t \quad (4)$$

The values of k_1 , q_e and k_2 were calculated (Table 1), it is seen that the values of equilibrium adsorption capacity (q_e) obtained in the case of pseudo-second-order model are more consistent with the experimental data, in addition to the higher R^2 values of the pseudo-second-order line than that of the pseudo-first-order line. Therefore, the adsorption reaction can approximate more favorable by the pseudo-second-order kinetic model.

Effect of initial metal concentration

The effect of initial Th(IV) species concentration on the adsorption using RI and RII resins was carried out using 50 mg resin with-

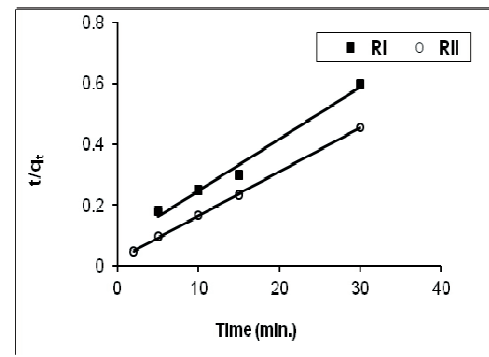


Fig. 9: Pseudo-second order plots for adsorption of Th(IV) ions on RI and RII resins

in series aqueous solutions (100 ml) containing Th(IV) ions ranged from 25 to 120 mg/l at pH 3.5. The contents were equilibrated at 20 °C for 15 min as the required time. Data presented on Figure (10) show that the adsorption capacity increased with increasing the initial Th(IV) concentration. At equilibrium, maximum adsorption capacities of Th(IV) ions were 50 and 64 mg/g for RI and RII resins respectively.

Effect of temperature

The effect of solution temperature on Th(IV) adsorption using the studied RI and RII resins was performed at a temperature ranged from 293-323 K. The obtained results

Table 1: Kinetic data for adsorption of Th(IV) ions using RI and RII resins

Resin	Pseudo-first order kinetics			Pseudo-second order kinetics			Experimental q_e
	q_1^{st}	k_1	R^2	q_2^{nd}	k_2	R^2	
RI	83.66	0.01	0.6469	58.82	0.004	0.9857	50
RII	39.09	0.07	0.9768	69.44	0.010	0.997	64

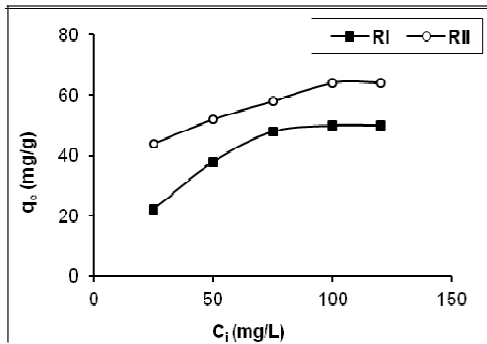


Fig. 10: Effect of Th(IV) ions initial concentration on adsorption by RI and RII resins

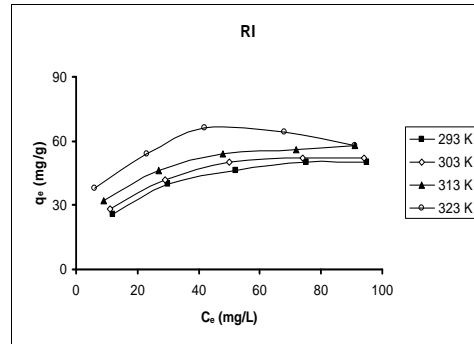


Fig. 11: Effect of temperature on the adsorption of Th(IV) ions by RI

(Figs. 11& 12) indicated that, the adsorption process is endothermic in nature. Increasing temperature from 293 to 323 °K resulted in increased Th(IV) ions adsorbed amount from 50 to 66 and from 64 to 88 mg/g for RI and RII resins respectively.

Adsorption isotherms

Adsorption isotherms describe the relationship between the q_e and C_e . The experimental data were tested at the Langmuir model (Eq. 5) to calculate the maximum capacity of the complete monolayer coverage of thorium on the resin surface (Abd El-Magied; 2016).

$$\frac{C_e}{q_e} = \frac{C_e}{q_{max}} + \frac{1}{K_L Q_{max}} \quad (5)$$

Q_{max} (Langmuir capacity, mg/g), and K_L (constant, L/mg) values were reported from

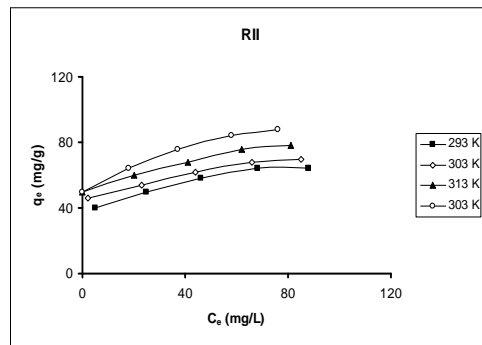


Fig. 12: Effect of temperature on the adsorption of Th(IV) ions by RII

Figures (13& 14) and given in Table (2). The calculated data show that K_L and Q_{max} values were increased as the reaction temperature increased which indicated a strong binding between Th(IV) ions and the active sites of the studied RI and RII resins.

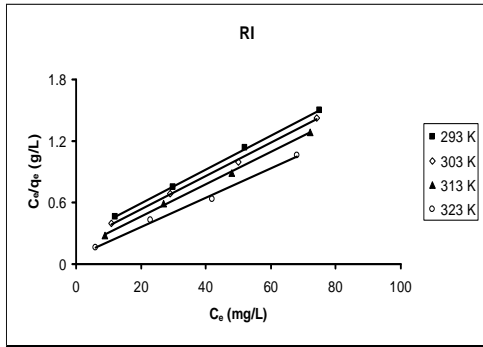


Fig. 13: Langmuir and plots for adsorption of Th(IV) on RI resin

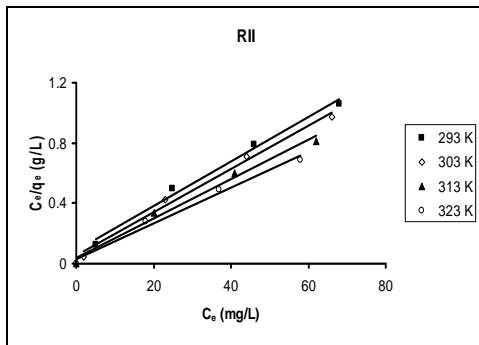


Fig. 14: Langmuir and plots for adsorption of Th(IV) on RII resin

The equilibrium parameter (R_L) can be calculated from the values of K_L according to Eq. 6:

$$R_L = \frac{1}{1 + K_L C_i} \quad (6)$$

The calculated R_L values lie between 0.006 and 0.23 indicated that, the suitability of the resin as Th(IV) adsorbents from aqueous solution.

Freundlich isotherm model was suggested for describing Th(IV) ions adsorption on RI and RII according to Eq. 7:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (7)$$

Plotting $\log q_e$ against $\log C_e$ was found to be linear. Langmuir's theoretical capacity (in addition to higher R^2 values) is more consistent with the experimental data than Freundlich isotherm indicated that the sorption reaction could be approximated more favorable by Langmuir model confirmed the monolayer coverage of Th(IV) ions on the used RI and RII.

The Thermodynamic parameters

Gibbs free energy is an important thermodynamic parameter since it enabled us to predict which reaction would occur spontaneously or not. Another important thermodynamic parameter in determining the type of adsorption was ΔH° . The thermodynamic parameters were determined using the classical thermodynamic Eq. 8, 9 (Abd El-Magied, 2016).

$$\ln K_L = \frac{-\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \quad (8)$$

Table 2: Langmuir and Freundlich parameters for Th(IV) adsorption on RI and RII at 20 °C

Resin	Freundlich parameters			Langmuir parameters			Experimental q_e
	N	K_f	R^2	Q_{max}	K_L	R^2	
RI	4.06	17.02	0.8346	70.92	0.0035	0.9834	50
RII	5.8	30.19	0.9644	65.36	0.26	0.9912	64

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (9)$$

Where R is the gas constant (8.314 J/mol.K), T is Kelvin temperature. The values of ΔS° , ΔH° and ΔG° were calculated, from the intercept and slope of the straight line, and were reported in Table (3).

Gibb's free energy (ΔG°) values were negative, which indicated the spontaneous nature of the adsorption process under the experimental condition. The positive values of ΔS° reflected the affinity of RI and RII resins towards Th(IV) species, while the calculated positive values of ΔH° indicated the endothermic nature of adsorption reaction.

Effect of solid/liquid phase ratio

The effect of the solid/liquid ratio on the adsorption process was achieved by varying the weight of beads from 25 to 200 mg in the adsorption medium (100 ml) while keeping other parameters constant. As seen on Figure (15), adsorption efficiencies of the resins were increased with increasing the solid/liquid ratio. The removal percentage (R %) of Th (IV) ions by RI and RII was calculated using Eq. 10.

$$R\% = \frac{(C_i - C_e)}{C_e} \times 100 \quad (10)$$

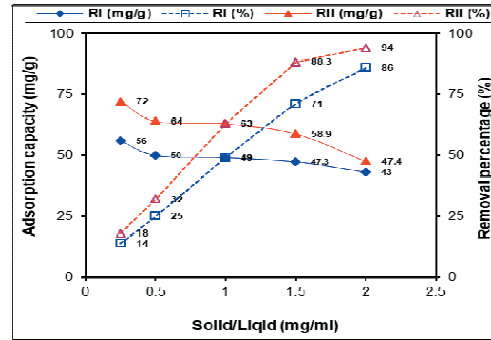


Fig. 15: Effect of the solid/liquid ratio of RI and RII resins on Th(IV) ions adsorption

Where C_i and C_e (mg/g) are the initial and equilibrium concentrations, respectively; R (%) is the removal percentage (Abd El-Magied, 2016).

The adsorption efficiencies were 86 and 94% at 100 ml adsorption solution with 200 mg (S/L; 2/1) of RI and RII resins respectively, while the adsorption capacities were decreased from 56 and 72 to 43 and 47.4 mg/g for RI and RII resins as the solid/liquid ratio increased from 1/2 to 2/1, respectively.

Desorption experiments

For the desorption process step, 0.5g of the loaded resins RI or RII after Th(IV) ions

Table 3: Thermodynamic parameters of Th(IV) ions adsorption using RI and RII resins

Resin	Temp. (K)	Thermodynamic parameters			
		ΔH° (kJ/mol)	ΔS° (KJ/mol. K)	$T \cdot \Delta S^\circ$ (kJ/mol)	ΔG° (kJ/mol)
RI	293	64.67	0.3046	89.25	-24.58
	303			92.29	-27.63
	313			95.34	-30.67
	323			98.39	-33.72
RII	293	31.64	0.196	57.43	-25.79
	303			59.39	-27.75
	313			61.35	-29.71
	323			63.31	-31.67

sorption was gently washed with distilled water and then was washed with 0.5 M HNO₃ to elute the adsorbed Th(IV) species. The elution efficiency attained about 92% over 3 cycles with a standard deviation of $\pm 3\%$. The eluted solution was subjected to the thorium precipitation step. The resin becomes regenerated and ready for the next use after gently washed with NaOH solution and water.

APPLICATIONS

The studied method, after optimization of all parameters, was examined for the adsorption of Th(IV) species from granite leach liquor solutions. Firstly, for this purpose, three granite rock samples were digested with 2M HNO₃ for 6 hours. The obtained slurries were filtered and then washed with hot distilled water. The obtained leach liquor solutions were analyzed for thorium determination. The measured thorium content in the leach liquors was; 18.3-24.9 mg/L while the rare earth oxide content was 22-29 mg/L. Secondly, the leach liquors were then treated with RI and RII resins for Th(IV) solid/liquid phase separation. The results show that; higher adsorption efficiencies towards Th(IV) species were 95 and 96% for RI and RII respectively, while rare earth ions content were (18-25%).

CONCLUSION

Glycidyl methacrylate resins functionalized using ethylene diamine and diethylene triamine were applied in the adsorption Th(IV) ions from nitrate solutions. The TGA curve permitted interpretations of the thermal stability of RI and RII and suggested RI is more stable than RII. At the solid/liquid phase ratio of 1/2 mg/ml, the maximum adsorption capacity towards Th(IV) ions on RI and RII were 50 and 64 mg/g respectively. The obtained results indicated the applicability of the resins for the Th(IV) adsorption from nitrate solutions by a spontaneous endothermic adsorption process. Finally, the applicability of the proposed method was examined successfully using some chosen granite rock

samples.

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إمتزاز أيونات الثوريوم الرباعي من محاليل حامض النيتريك باستخدام راتينجات أمين جلاسيديل ميثا أكريلات

محمود عثمان عبد المجيد، السيد عبد الهادي مناع، محمد نوح كريم، محمد عبده يوسف و إكرامي محمد الدسوقي

تناول هذا البحث دراسة فصل أيونات الثوريوم الرباعي عن محاليل حمض النيتريك باستخدام راتينجات جلاسيديل ميثاكريلات/ ديفينيل بنزين/أمين والمسماة RI و RII. في البداية تمت تحضير وتوصيف الراتنجات وكذلك تم دراسة الثبات الحراري. تم استخدام الراتنجات المحضرة لفصل أيونات الثوريوم من المحاليل الحمضية ودراسة العوامل المؤثرة علي عملية الفصل. وقد أشارت النتائج إلي زياده قدره الراتنجات علي فصل أيونات الثوريوم مع زيادة درجه حموضة الوسط وكان الحد الأقصى لإمتصاص أيونات الثوريوم عند درجة حراره الغرفه ٥٠ و ٦٤ ملجم/ جم لكل من الراتنجات RI و RII عند درجة حموضة ٣,٥ على التوالي. وقد لوحظ أيضا ان زيادة درجة الحرارة لها تأثير إيجابي علي عملية الفصل حيث إرتفع معدل إمتصاص ايونات الثوريوم للراتنجات RI و RII من ٥٠ إلى ٦٤ ومن ٦٦ إلى ٨٨ ملجم/ جم بزيادة حرارة المحلول محل الدراسة من ٢٩٣ إلى ٣٢٣ كلفن على التوالي ودلت النتائج إلى قابلية تطبيق الراتنجات RI و RII لإمتصاص أيونات الثوريوم من المحاليل المختلفة. أخيراً تم تطبيق الراتنجات لفصل أيونات الثوريوم بكفاءه عالية من محاليل النترات لصخور جرانيتية مختلفة