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The removal of uranium and thorium from their aqueous solutions via glauconite

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ABSTRACT

The sorption behavior of uranium and thorium from their aqueous solutions by local Egyptian Bahariya oases glauconite has been investigated by the batch technique. The parameters that affect the sorption of uranium and thorium on glauconite such as pH, contact time, their initial concentration, glauconite mass, and temperature have been studied. Sorption experiments were expressed by Langmuir and Freundlich isotherms and the results demonstrated that the sorption of uranium and thorium on glauconite correlated well with Freundlich isotherm. Kinetics studies showed that the sorption followed pseudo–first-order kinetic model with an activation energy (E_a) of 8.8 kJ/mol. Thermodynamic parameters such as ΔH°, ΔS°, and ΔG° indicated that the sorption of uranium and thorium on glauconite was endothermic.

Keywords: Sorption; Uranium; Thorium; Glauconite; Uptake

1. Introduction

Many activities dealing with radioactive materials produce low, intermediate, and high-level radioactive wastes that require advanced treatment [1–3]. The removal of radionuclide and toxic heavy metals such as uranium and thorium from aqueous waste solutions seems to be a significantly useful subject for environmental control [4]. In view of the extensive usage of uranium and thorium for various industrial purposes and their toxicity, their removal is an important aspect especially as to the environment protection. Both U(VI) and Th(IV) are extremely mobile and, once entered the living bodies, provoke the internal exposure (especially due to the γ-active decay products), having, as a final result, the appearance of cancer [5]. The major use of uranium and thorium as nuclear fuel for the production of nuclear energy in a nuclear reactor is well known [6]. Unlike uranium, thorium and its compounds and alloys find extensive application [7].

Many processes have been proposed for uranium (VI) and thorium (IV) removal from industrial wastewaters and radioactive wastes. Chemical precipitation, membrane processes, ion exchange, and adsorption are the most commonly used methods [8–11]. Adsorption represents an effective and economic technique usually applied for the removal, recovery, and

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Table 1
Major chemical constituents of glauconite

	Constituents										
	SiO_2	Al_2O_3	FeO (total)	P_2O_5	Na_2O	CaO	MgO	K_2O	MnO	Cl^-	LOI
Mass %	29.00	23.50	16.90	0.37	0.17	0.04	4.40	23.50	0.05	0.2	1.87

recycling of numerous elements from wastewater, with the advantage that low amounts of substances may be separated from large volumes of solution. A number of materials, such as activated carbon, zeolite, olivine rock, coir pith, smectites, kaolinite, bentonite, montmorillonite, biomass, goethite, and polymeric materials, have been used for the adsorption of uranium (VI) and thorium (IV) [10–12].

Glauconite, a clay mineral, is a natural three-layer sheet aluminosilicates occurring primarily in marine sediments. It contains multivalent iron, aluminum, and magnesium ions confined between two flat silicate layers that also contain OH^- groups; the triple layers alternating with a layer of water molecules and alkaline earth metals, most notably potassium. A general formula may be postulated as [13] $(K, Na, Ca)_{1.2–2.0}$ $(Fe^{3+}, Al, Fe^{2+}, Mg)_{4.0}$ x $[Si_{7–7.6}\ Al_{1–1.4}O_{20}]$ $(OH)_4\ nH_2O$.

It is anticipated that glauconite has metal-adsorbing ability where NaCl- and NaOH-treated glauconite-bearing sand was used to remove Zn(II), Pb(II), Cd(II), and ammonium [14]. Greensand containing 80% glauconite was used to remove Cd, Co, Cu, Pb, Mn, Ni, Ag, and Zn [15]. Sorption of U(VI) on glauconite was also examined [16,17], while for thorium no previous sorption studies were carried out.

The main objective of this study was to investigate the sorption of uranium and thorium from aqueous solutions on local Egyptian Bahariya oases glauconite as a function of various experimental parameters. Additionally, isotherms, kinetics, and thermodynamics characteristics of the sorption process of the U(VI) and Th(IV) from their aqueous solutions on glauconite were also investigated.

2. Experimental

2.1. Materials

2.1.1. Glauconite

Glauconite was brought from Bahariya Oases, Egypt. It was ground and screened to a particle size (0.2–0.3 mm). The received glauconite was placed in a glass container and was kept in a desiccator all the time of the experiments. The chemical constituents of glauconite (Table 1) were identified using X-ray fluorescence (PANalytical Axios advanced, The Netherlands). The constituents' phases of glauconite were identified by an X-ray diffraction analysis (XRD brucker axs D8 advance, Germany) with CuKα radiation (1.5406 Å) with a typical scanning beginning at 2θ equal to 20–80° and a scan rate of $20\,min^{-1}$. X-ray diffraction pattern of glauconite is shown in Fig. 1.

2.1.2. Reagents

Standard individual solutions for U(VI) and Th(IV) (1,000 mg/L) were prepared from $UO_2(NO_3)_2{\cdot}6H_2O$ and $Th(NO_3)_4{\cdot}5H_2O$ (Riedel-de Haen AG, Germany) by dissolving 2.10 and 2.46 g, respectively, in 1 L of double distilled water. Arsenazo (III) was obtained from Aldrich. Co., Germany. All other reagents used were of analytical reagent grade. All the solutions used were prepared in doubled-distilled water.

2.2. Sorption experiments

The sorption experiments were studied by a batch technique. In the experiments, glauconite was individually shaken with each element solution at various experimental conditions. Thiel buffer [18] was used to maintain the pH of the solutions in the pH range (2–6) ± 0.1. Separation of solid phase from liquid was done by centrifuging at 4,000 rpm for 15 min. After equilibrium, the concentration of uranium and thorium was determined spectrophotometrically using

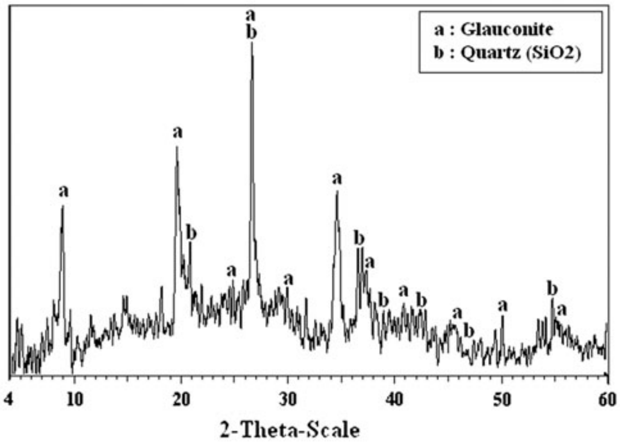

Fig. 1. X-ray diffraction of glauconite.

Arsenazo-III (0.05%, w/v) as a complexing agent at 655 and 659 nm, respectively, against reagent blank [19]. The determination was made employing a Shimadzu UV-vis160a Spectrophotometer (± 0.005).

Uptake percentage (%*E*) and distribution constant K_D (mL/g) were calculated using Eqs. (1) and (2), respectively:

$$\%E = \frac{C_o - C_e}{C_o} \times 100 \qquad (1)$$

$$K_D = \left(\frac{C_o - C_e}{C_e}\right)\frac{V}{m}\,(\text{mL/g}) \qquad (2)$$

where C_o and C_e are the initial and equilibrium concentrations in the solution (mg/L), respectively. V is the volume of the aqueous solution (mL) contacted with glauconite and m is the mass of glauconite in grams.

3. Results and discussion

3.1. Sorption factors

The factors, which may affect the sorption of uranium and thorium on glauconite such as contact time, temperature, pH, U(VI) and Th(IV) initial concentration, sorbent mass and temperature, were investigated.

3.1.1. Effect of contact time

Fig. 2 shows the effect of contact time on U(VI) and Th(IV) sorption on glauconite. The results showed that sorption was fast and the uptake percentage increased with time till the equilibrium reached maximum within 5 min of shaking for both elements and remained constant. According to the above results, 6 min was adopted for the subsequent experiments to make sure that equilibrium was reached.

3.1.2. Effect of initial pH

The results of the influence of pH on the sorption of uranium and thorium on glauconite are shown in Fig. 3. The results demonstrated that the sorption of U(VI) and Th(IV) onto glauconite increased sharply with increasing pH, then it reached maximum value at pH 4. Cations hydrolysis, accompanied by the formation of some insoluble or soluble species, seems to be an important element of this variation. Through hydrolysis, uranium may form a series of aquocomplexes, such as $UO_2(OH)^+$, $(UO_2)_2(OH)_2^{2+}$ and $(UO_2)_3(OH)_5^+$ [20]. The ratios of these species depend on the uranium ions concentration and the solution pH. Over the 1.3–4.0 pH interval the predominant species are $(UO)_2^{2+}$, $UO_2(OH)^+$, $(UO_2)_2(OH)_2^{2+}$ and $(UO_2)_3(OH)_5^+$ [21]. For Th(IV) which is the least hydrolyzed tetrapositive ion [22], the distribution diagram for thorium species was calculated [23] at ionic strength of 0.1 mol/L from the hydrolysis constants. It was found that at pH 3, Th(IV) is the predominating species (⩾88%), $Th(OH)^{3+}$ is less than 12%; at pH 4, $Th(OH)^{3+}$ is less than 40%, $Th(OH)_2^{2+}$ is less than than 28%, $Th(OH)^{3+}$, and $Th(OH)_4$ are less than 12% [23].

It is clear that at pH ⩾ 3, the hydrolysis products and the precipitation begin to play an important role in the sorption of U(VI) and Th(IV) [24–26].

Low uptake at low pH values is most probably due to the protonation of the active sites in glauconite, which inhibits their binding ability towards the U(VI) and Th(IV) [27]. In addition, as pH increases, surface positive charge decreases; this would result in lower columbic repulsion of the sorbed metal ions [28].

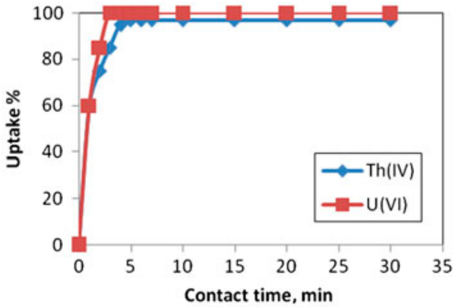

Fig. 2. Variation of the uptake percentage with contact time for U(VI) and Th(IV) sorption on glauconite. Operating conditions: 50 mL solution, pH = 4, $[U^{6+}]$ or $[Th^{4+}]$ = 50 mg/L, and 0.04 g glauconite.

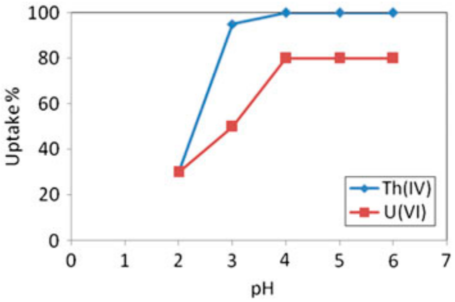

Fig. 3. Variation of the uptake percentage with pH for U(VI) and Th(IV) sorption on glauconite. Operating conditions: 50 mL solution, $[U^{6+}]$ or $[Th^{4+}]$ = 100 mg/L, 0.04 g glauconite, and contact time = 6 min.

Moreover, the observed reduction in the percentage of U(VI) and Th(IV) uptake on glauconite at low pH by the sorbent indicates that sorption of U(VI) and Th(IV) is most probably due to an ion exchange process [29].

3.1.3. Effect of the initial concentration of U(VI) and Th(IV)

Fig. 4 illustrates the effect of the initial concentration of U(VI) and Th(IV) on their sorption on glauconite by varing their initial concentration from 50 to 600 mg/L. The inverse correlation between U(VI) and Th(IV) initial concentration and uptake percentage reflects the greater partitioning of these elements into the solid phase up to 400 mg/L for Th(IV) and to 500 mg/L for U(VI), respectively. With increasing U (VI) and Th(IV) concentration, the uptake percentage stabilizes, owing to saturation of the glauconite surface. These results are similar to those previously reported for the sorption of both ions on peat moss [30]. These results indicate that energetically less favorable sites become involved with increasing U(VI) and Th(IV) concentration in the aqueous solution.

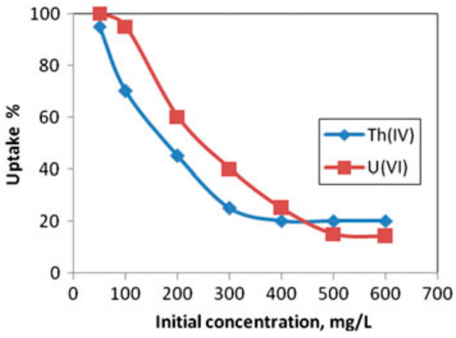

Fig. 4. Variation of the uptake percentage with the U(VI) and Th(IV) initial concentration for their sorption on glauconite. Operating conditions: 50 mL solution, pH = 4, 0.04 g glauconite, and contact time = 6 min.

3.1.4. Effect of sorbent mass

Effect of sorbent mass on the sorption process of Th(IV) and U(VI) was studied. As illustrated in Fig. 5, the sorption of Th(IV) and U(VI) increased with increasing amount of glauconite up to 0.03 g, beyond which the sorption capacity does not change with the sorbent mass. Increasing number of sorbent particles in the solution allows more uranium and thorium ions to interact with the reaction sites.

3.1.5. Effect of temperature

Effect of temperature on the sorption process of U (VI) and Th(IV) is shown in Fig. 6. It was found that the uptake percentage of both metal ions was increased with the increase of temperature. This may be due to the increase of the number of reacting molecules having excess activation energy which leads to the increase of sorption rate and the rate of mass transfer of the diffusion [31].

3.2. Sorption isotherms

In order to understand the sorption behavior of glauconite for U(VI) and Th(IV), the equlibrium data were evaluated according to different sorption isotherms, namely, Langmuir and Freundlich isotherm models over a range of 100–500 mg/L for both elements.

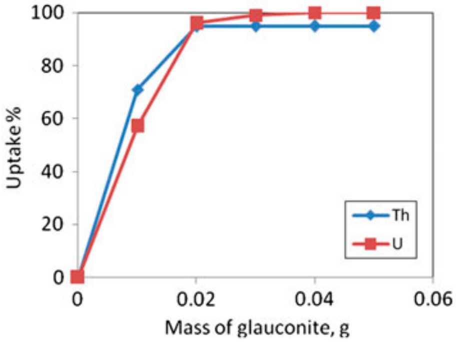

Fig. 5. Variation of the uptake percentage with the sorbent mass for U(VI) and Th(IV) sorption on glauconite. Operating conditions: 50 mL solution, $[U^{6+}]$ or $[Th^{4+}]$ = 50 mg/L, pH = 4, and contact time = 6 min.

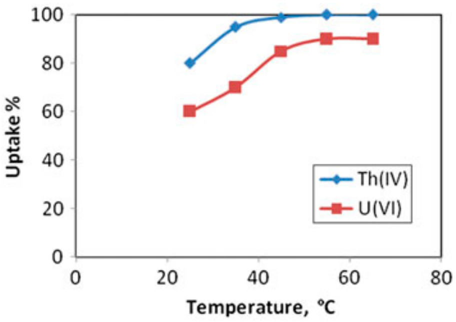

Fig. 6. Variation of the uptake percentage with the temperature for U(VI) and Th(IV) sorption on glauconite. Operating conditions: 50 mL solution, $[U^{6+}]$ or $[Th^{4+}]$ = 50 mg/L, pH = 4, 0.01 g glauconite, and contact time = 6 min.

3.2.1. Langmuir isotherm

The widely used Langmuir isotherm has been applied to many sorption processes and is expressed as in Eq. (3):

$$\frac{C_e}{q_e} = \frac{1}{bQ_e} + \frac{C_e}{Q_e} \tag{3}$$

where C_e is the equilibrium concentration of U(VI) and Th(IV) in solution (mg/L); q_e is the amount of solute sorbed per unit mass of glauconite at equilibrium (mg/g); and Q_e (mg/g) and b (L/mg) are the Langmuir constants related to monolayer sorption capacity and free energy of sorption, respectively. A plot of C_e/q_e versus C_e would result in a straight line with a slope of ($1/Q_e$) and intercept of $1/bQ_e$ as shown in Fig. 7. The values of the slopes and intercepts of the plots are presented in Table 2.

The Langmuir model considers that the sorbent surface is homogeneous with identical sites in terms of energy. The values of correlation coffiecients (R^2) indicate that the langmuir isotherm model does not fit the sorption of U(VI) and Th(IV) on glauconite.

3.2.2. Freundlich isotherm

The well-known Freundlich isotherm is often used for heterogenous surface energy systems. A linear form of this expression is as Eq. (4).

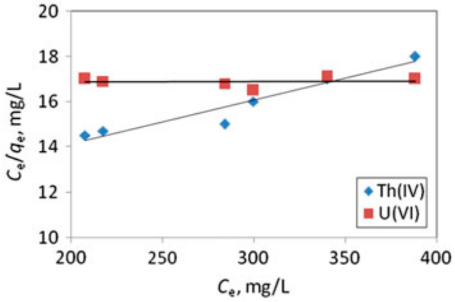

Fig. 7. Langmuir sorption isotherm of U(VI) and Th(IV) sorption on glauconite.

$$\log q_e = \log k + \frac{1}{n}\log C_e \tag{4}$$

where q_e is the amount of solute sorbed per unit mass of glauconite at equilibrium (mg/g) and C_e is the equilibrium concentration of U(VI) and Th(IV) in solution (mg/L). k and n are constants characteristics of the system. Log k and $1/n$ are the Freundlich constants related to sorption capacity and sorption intensity of the sorbent, respectively. Fig. 8 shows the plot of log q_e versus log C_e. Freundlich constants together with correlation coffiecients are presented in Table 2. The linear form of the plot over the entire concentration indicates the applicability of the freundlich model to the sorption of U(VI) and Th(IV) on glauconite. The values $1/n$ of indicate the favorable sorption of U(VI) and Th(IV) on glauconite. This model was also found to fit the sorption of U(VI) on date pits [32] and the sorption of Th(IV) onto activated carbon from olive stones [33].

3.3. Sorption kinetics

For simplicity, the relation between the uptake percentage and time at different temperatures, 303, 313, 323, and 333 K, onto glauconite was investigated for uranium. The results obtained are represented in Fig. 9. It is obvious that the sorption rate was very high at the initial time and then the rate decreased. The analysis of the curves relating the uptake percentage and time shows that each curve has three different slopes, indicating three different values of sorption rates. The first value was high, while the second was somewhat slower and the third was the slowest one.

The rate constant for the retention step was evaluated in the light of the Lagergren pseudo-first-order rate (Eq. 5) [34]:

$$\log(q_e - q_t) = \log q_e - \left(\frac{kt}{2.303}\right) \tag{5}$$

where q_e and q_t are the sorbed concentration at equilibrium and at any time t, respectively; and k is the overall rate constant of first-order sorption.

Table 2
Isotherms constants and values of correlation coefficient (R^2) for sorption of U(VI) and Th(IV) on glauconite

Element	Freundlich isotherm			Langmuir isotherm		
	$1/n$	log k	R^2	Q_e (meq/g)	b (L/mg)	R^2
Th(IV)	0.5357	2.6518	0.9875	58	0.0016	0.0358
U(VI)	0.9362	1.8497	0.9956	1,111	5.3861	0.5720

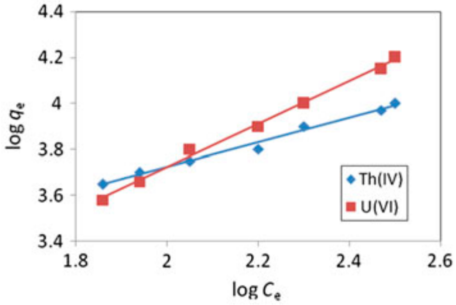

Fig. 8. Freundlich sorption isotherm of U(VI) and Th(IV) sorption on glauconite.

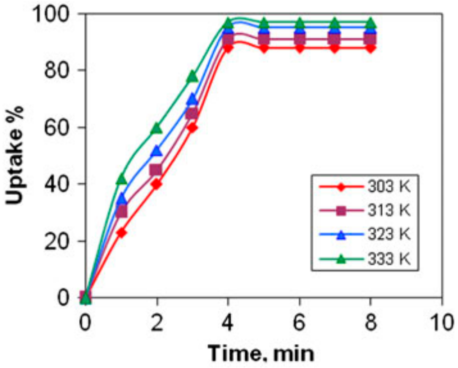

Fig. 9. Variation of the uptake percentage of U(VI) with time at different temperatures. Operating conditions: 50 mL solution, $[U^{6+}] = 50$ mg/L, pH = 4, and 0.01 g glauconite.

Table 3
The calculated parameters of the pseudo-first-order model for U(VI) sorbed on glauconite at different temperatures

Temperature, K	First-order kinetic parameters		
	k (min^{-1})	R^2	E_a (kJ/mol)
303	0.0510	0.9918	8.8
313	0.0576	0.9889	
323	0.0645	0.9944	
333	0.0691	0.9920	

The calculated values of k and E_a with the values of the linear correlation coefficients (R^2) of each plot are presented in Table 3. The studies show that the pseudo-first-order kinetic model fit the data for the sorption process since the values of correlation factor R^2 were high.

The activation energy of the sorption process can be determined using Arrhenius equation. It can be calculated from the slope of a plot of log k and $1/T$, since the slope equals to $(-E_a/2.303R)$. Since the activation energy is in the range of 0–40 kJ/mol, the sorption of uranium and thorium on glauconite is physically one [10].

3.4. Thermodynamic parameters

The sorption of U(VI) and Th(IV) on glauconite was carried out at different temperatures, 303, 313, 323, and 333 K, for better understanding the mechanism of the sorption process. The thermodynamic parameters ΔS° and ΔH° were calculated using van't Hoff's equation (Eq. 6):

$$\ln K_D = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \tag{6}$$

where K_D is the distribution coefficient (mL/g), ΔS° is the standard entropy (J/mol K), ΔH° is the standard enthalpy (kJ/mol), T is the absolute temperature (K), and R is the gas constant (8.314 J/mol K). The values of ΔH° and ΔS° were respectively calculated from the slopes and intercepts of plots of ln K_D versus $1/T$. The values of standard Gibbs free energy ΔG° (kJ/mol) were calculated using Eq. (7):

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \tag{7}$$

The values of ΔH°, ΔS°, and ΔG° are listed in Table 4. The positive value recorded for ΔH° for the process confirms the endothermic nature of the process, which indicates an existence of a strong interaction between glauconite and U(VI) and Th(IV), while the positive entropy of sorption ΔS° reflects the affinity of

Table 4
Thermodynamic parameters for sorption of U(VI) and Th(IV) on glauconite

Element	ΔH° (kJ/mol)	ΔS°(kJ/mol K)	ΔG°(kJ/mol)			
			303 K	313 K	323 K	333 K
Th(IV)	13.34	0.513	−142.1	−147.23	−152.34	−157.48
U(VI)	64	0.273	−18.7	−21.45	−24.18	−26.9

glauconite toward U(VI) and Th(IV). The estimated values of ΔG° point to the feasibility of the sorption process of U(VI) and Th(IV) on glauconite and its spontaneous nature without an induction period. The decrease in ΔG° values with the increase of temperature indicates more efficient sorption at higher temperature.

4. Conclusions

- The sorption of uranium and thorium from aqueous solutions on glauconite was pH-dependent and the maximum sorption of both elements occurred at pH 4.
- The equilibrium data have been analyzed using Langmuir and Freundlich isotherms. The Freundlich isotherm was demonstrated to provide the best correlation.
- The sorption of U(VI) and Th(IV) on glauconite was found to follow pseudo-first-order kinetics with the activation energy of 8.8 kJ/mol. The thermodynamic parameters ΔH°, ΔS°, and ΔG° values of the sorption onto glauconite showed endothermic heat of sorption, which favored at high temperatures. The positive entropy value is an indication of the probability of favorable nature of sorption and the process is spontaneous.
- The experimental results indicated that the glauconite can be effectively applied for the removal of U (VI) and Th(IV) from aqueous solutions.
- The present study recommends the application of glauconite in the treatment of contaminated wheels with radioisotopes of lanthanides and actinides. Glauconite can be used as scavengers in drainage waste water treatment in agriculture, industrial, and radio-research laboratories.

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