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Determination of Distribution Coefficients of Uranium and Thorium on Bedrock of Anarak Nuclear Waste Repository Using Batch Sorption Experiments

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ABSTRACT

One of the key influencing parameters in the safe management of nuclear waste repositories is the distribution coefficient (K_d) of radionuclides on bedrock. In this study, (K_d) of uranium and thorium ions in intact bedrock were determined using batch experiments on crushed bedrock at seven different particle size fractions as well as three different initial concentrations of ions. Sorption experiments have been performed on crushed bedrock prepared from intact drill core samples and an aqueous solution containing desired ions from the local water (LW) near Anarak Nuclear Waste Repository. The results showed that both factors of the initial ions concentration as well as the size of the crushed bedrock particles significantly affect the value of the distribution coefficient so that (K_d) value increases with decreasing particle size and increasing ion concentration. Also, (K_d) calculated at three studied concentrations showed that the difference in the (K_d) values in smaller particles is large but decrease with increasing particle size and (K_d) curves for two concentrations of (100 and 10) ppm overlap at the end of the chart. Therefore, it can be concluded that for accurate calculation, it is better to use large particles and low concentrations of ions to determine (K_d) in intact bedrock. In this report, different mechanisms including chemical interactions, physical adsorption, and ion exchange were presented for ion adsorption by bedrock. The proposed mechanisms were related to the type of metal speciation of ions in the solution. Based on the obtained data, the preferred mechanism to describe the adsorption of uranium and thorium ions is composed of chemical interactions and physical adsorption.

Keywords: Distribution coefficients, Anarak repository, Nuclear waste, Batch sorption.

1. Introduction

Anarak Nuclear Waste Repository is designed for disposing of low-level radioactive

waste generated in nuclear power plants and fuel cycles in Iran. Based on Iran Nuclear

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Regulatory Authority (INRA) requirements, safety assessment should be conducted in several licensing stages including site selection, construction, operation, site closure, and Post-closure stages [1]. One of the key parameters in safety assessment is the distribution coefficient (Kd) of radionuclides. This has a significant contribution to radionuclide transport in the geosphere. The distribution coefficient reflects the chemical properties of rock and the magnitude of sorption in considered conditions and depends strongly on the type and mineral composition of bedrock, the chemical composition of the local water of the studied geographical area, and the type of radionuclide in question. The safety assessment also assumes equilibrium and reversibility of the sorption process.

Default estimates of Kd presented in articles, textbooks, and technical documents involve a wide range of radionuclides in order from ten to the power of 4. These radionuclides impose huge uncertainties in public dose assessment. Up to now, the IAEA has published several documents to address this uncertainty in safety assessment [2-6]. However, despite all the efforts made, the uncertainties associated with the solid-liquid distribution coefficient (Kd) in some rock types remain. According to IAEA recommendations, site-specific Kd measurements should be performed in each nuclear waste repository to enhance confidence building and decrease associated uncertainties [7]. To date, numerous studies have been carried out on the influence of pH, rock type, grain size, temperature, height of the column, and radionuclide concentration on distribution coefficients [8-14]. Usually, the distribution coefficients of different minerals and rocks can be determined by static batch experiments with broken rock. Crushing, however, increases the specific surface area of rock in comparison to intact rock, and the results may overestimate the distribution coefficient of intact rock. It has been found that distribution coefficients

obtained from crushed rock can be significantly higher than those obtained using intact rock. Previously, attempts have been made to determine the distribution coefficients of intact rock using through-diffusion experiments, electromigration sorption experiments, and in situ experiments. For example, electromigration as a widely used technique is based on enhancing the migration of ions into intact rock. This is done by applying electrical potential to a drill core sample. Due to the enhancement, longer rock cores can be collected than in traditional diffusion experiments. Therefore, effects arising from sample preparation, e.g. drilling and sawing, are reduced. In general, the method is faster than traditional diffusion experiments used for determining the effective diffusion coefficient. To perform electromigration experiments, the method has been further developed. Furthermore, the samples' porosities were determined using water saturation and immersion methods [15]. However, these experiments are time-consuming, especially for strongly sorbing nuclides, and in the through-diffusion method, only a limited sample size can be applied, which may cause problems due to sample preparation, drilling, and sawing. Due to these issues, it would be beneficial if batch sorption experiments could be conducted to determine reliable distribution coefficients for intact rock. This would enable us to estimate them. However, it is not yet understood how batch sorption experiments could be converted to intact rock. Safety assessment solved this issue using a conversion factor for batch sorption data. Conversion factors have been obtained by comparing sorption values for crushed and intact rock samples. Another option is to compare their specific surface areas. In both cases, determining values for intact rock samples is laborious and time-consuming. Since data do not exist for all systems, conservative values in safety assessments are used for conversion factors. The main aim of this study

was to test if a batch sorption experiment involving seven different particle size fractions from 0.10 to 2.7 mm as well as three different initial concentrations can be applied to evaluate the K_d of intact crystalline rock samples for Uranium and Thorium instead of performing time-consuming diffusion and electromigration experiments [16].

2. Material and methods

The bedrock sample was obtained from inside the Anarak Nuclear Waste Repository trench in Nain County, Isfahan Province, Iran. $UO_2(NO_3)_2 \cdot 6H_2O$ and $Th(NO_3)_4 \cdot 5H_2O$ were purchased from Sigma Aldrich company. The water required for batch adsorption experiments of the studied ions was prepared from groundwater near the Anarak Nuclear Waste Repository. For batch tests, high-precision scales, shakers, and centrifuges from Sartorius (Germany), Memmert (Germany), and Mistral (USA) Company were used. Uranium and solution were measured with an ICP-AES, optima 7300 DV (USA). XRD, Philips pw 1800 (Netherlands), and Olympus BX60 microscope were utilized for the determination of the main minerals in the bedrock and Petrographical studies (including general recognition of mineralogy and textures).

2.1 Bedrock sampling

Bedrock samples were selected from drill cores up to 4 meters. The location of the core samples harvested is shown as TR3 on the map in Figure 1. The geographical coordinates of the point are presented in Table 1.

Table 1. Geographic coordinates of sampled bedrock (TR3).

Longitude UTM	Latitude UTM
729800	3694745

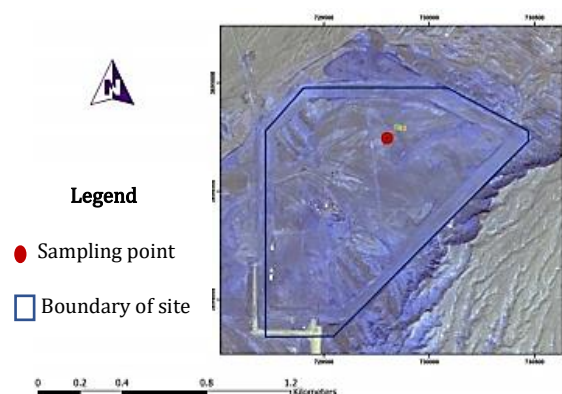


Fig. 1. Action of rock sampling for distribution coefficient analyses.

2.2 Bedrock and groundwater characteristics

The rock samples have been crushed and screened and seven different (0.1, 0.3, 0.5, 0.7, 1, 1.7, and 2.3 mm) particle size fractions have been used in the experiments. For mineralogical studies including petrography and bedrock structural characteristics, 3 and 2 samples for thin section and XRD were taken respectively. Macroscopic evidence of the hand specimens shows that they are sedimentary rocks composed of grains with a diameter of less than 2 mm. Petrography of thin sections confirms that the hand specimens are sandstone type with a matrix content of more than 15% (Figures 2 A, B). According to sandstone classification, the word "wacke" is used for the nomenclature of sandstones with more than 15% clay matrix [17-18]. In this classification, sandstones that contain more rock fragments than feldspar are called "lithic greywacke" and if feldspar is more than rock fragments, it is called "feldspathic greywacke" (Figure 3).

According to the effect of groundwater on the heavy metal distribution coefficient, groundwater was prepared from the Waste Repository Anarak Nuclear Waste Repository. It was filtered through a 0.45- μ m polycarbonate membrane and the numbers of ions and anions were determined by ion chromatographic analysis (IC), inductively coupled plasma (ICP-AES), and atomic absorption spectrometry (AAS) before being used in distribution coefficient measurement.

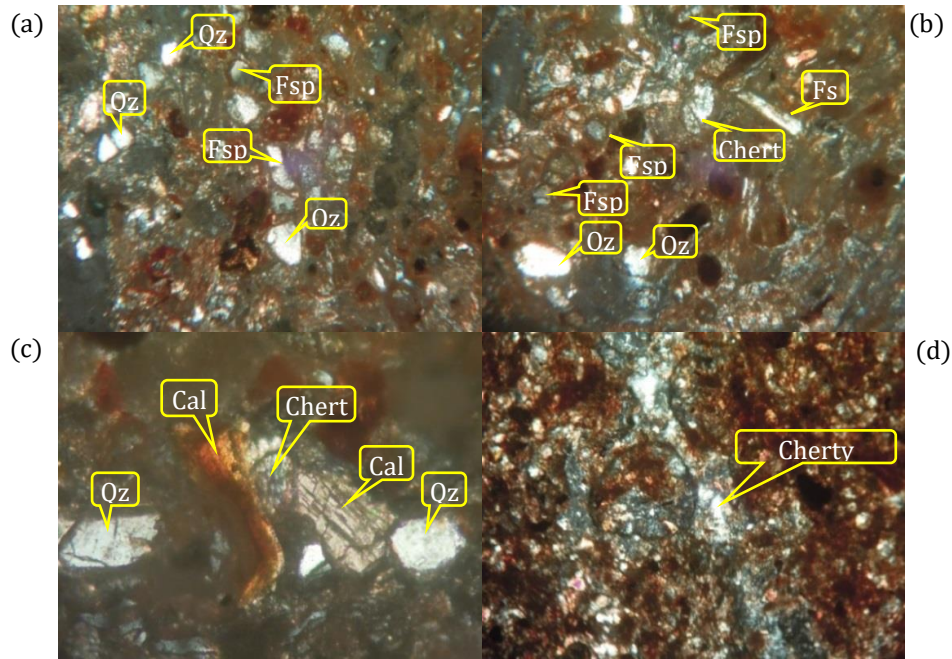


Fig. 2. Photomicrograph transmitted-light (in XPL): (a) quartz and feldspar within a clay matrix (under 10x), (b) quartz and chert within a clay matrix (under 10x), (c) calcite along with quartz and chert within a clay matrix (under 20x), (d) secondary chert cement within a clay matrix (under 4X). (Abbreviation: Qz=quartz, Fsp=feldspar, Cal=calcite).

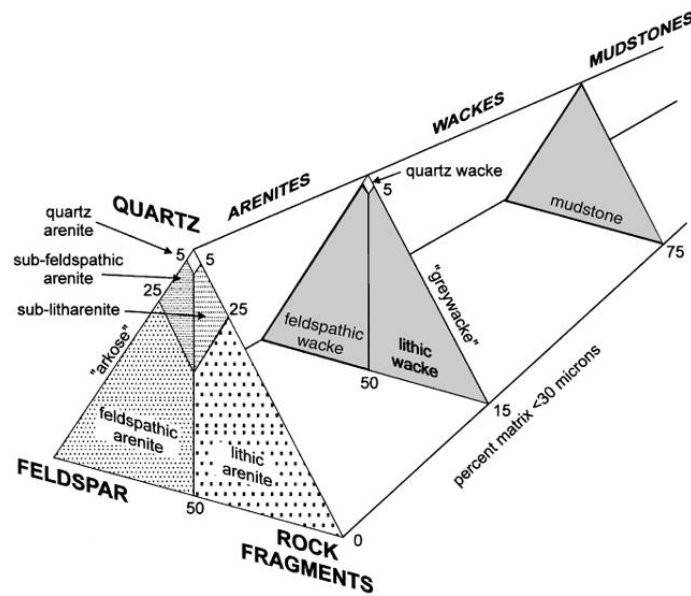


Fig. 3. Classification of Sandstones [17].

2.3 Equilibrium test of bedrock with groundwater or cold wash

In order to calculate the correct and accuracy of K_d , before performing experiments with solutions containing ions, the tests must first be performed with groundwater for a week. After this time, the samples are collected and heated at 80 °C for 48 hours to dry and prepare for the

next step. This is done to create equilibrium between the ions in the bedrock structure and the groundwater. Because the calculation of K_d is close to real values if it is in a competitive environment with ions in water. In fact, cation exchange will first take between the ions in the groundwater and the bedrock and then the exchanged ions will be replaced with the

desired ions in the study solution in the next step.

2.4 Distribution coefficient and adsorption capacity determination

The distribution coefficients and adsorption capacity of metal ions in crushed bedrock were obtained by the batch method described below. To conduct this experiment, 1g of crushed bedrock samples at different mean grain sizes (0.1, 0.3, 0.5, 0.7, 1, 1.7, and 2.3 mm) was suspended in 50 mL of metal ion solutions at different concentrations of 10, 100 and 1000 mg/l for a week. The mixture was continuously shaken in a shaking batch at room temperature (25°C) followed by centrifugation at 2000 rpm for 10 minutes. The number of metal ions loaded on the sorbent particles is calculated by conducting a mass balance on the solute. This is done before and after the test using ICP. The adsorption capacity and distribution coefficient were calculated using Equation [19]

$$q_e = \frac{(C_0 - C_e)V}{M} \quad (1)$$

$$K_d = \frac{C_0 - C_e}{C_e} * \frac{V}{M} \quad (2)$$

Where C_0 and C_e are the initial and final concentration of the ions, V is the volume of the initial solution (ml) and M is the mass of the crushed bedrock in gram.

3. Results and discussion:

3.1 Bedrock and groundwater analysis

So far, various studies have been performed to determine radionuclide and heavy metal distribution coefficients on bedrock samples. The results showed that values with different ranges depend on the type of bedrock and the chemical composition of groundwater. Therefore, in this study, according to EPA instructions, the bedrock and groundwater of the Anarak Nuclear Waste Repository were analyzed. Petrography of thin sections showed that the clay matrix makes up about 20 to 30% of the rock volume (Figures 2A, B) and this is confirmed by XRD analysis (Figure 4 and Table 2). The main minerals found in thin sections include quartz, feldspar, and calcite in a matrix of iron-bearing (red) clay minerals (Figures 2B, C). The quantitative XRD analysis results showed two types of feldspars, including albite (15-17%) and microcline (6-8%). In addition to the minerals observed in petrographic studies, semi-quantitative XRD analysis shows montmorillonite (29-32%), illite (8-9%), chlorite (5%), and gypsum (2-3%) minerals. Based on microscopic studies and XRD analysis results, the amount of quartz and feldspar (albite and microcline) is almost equal. Therefore, the sandstone can be called "quartz-bearing feldspathic greywacke" or "quartz feldspathic greywacke". In this rock, in addition to the clay matrix, carbonate (calcite) cement and secondary chert cement are also formed.

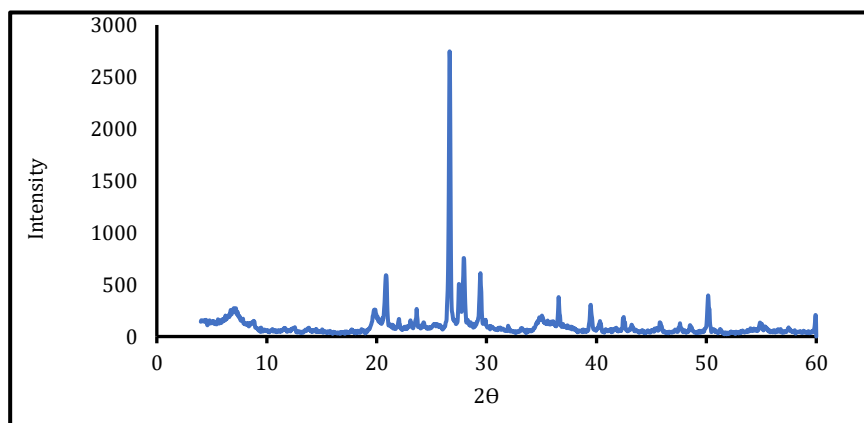


Fig. 4. Results of quantitative XRD.

Table 2. Results of quantitative XRD.

Sample	Phase(s)	Phase(s)
HA-1398-ANR-002	Montmorillonite (13-0135) = 32%	Calcite (05-0586) = 6%
Az : 22584-127191	Ca _{0.2} (Al,Mg) ₂ Si ₄ O ₁₀ (OH) ₂ ·xH ₂ O	CaCO ₃
Date : 2/3/2020	Quartz (33-1161) = 24%	Muscovite - illite (26-0911) = 8%
	SiO ₂	KAl ₂ Si ₃ AlO ₁₀ (OH) ₂
kV = 40	Albite (09-0466) = 15%	Chlorite (29-0701) = 5%
mA = 30	NaAlSi ₃ O ₈	(Mg,Fe) ₆ (Si,Al) ₄ O ₁₀ (OH) ₈
Ka. = Cu	Microcline (19-0932) = 6%	Gypsum (33-0311) = 2%
Fil. = Ni	KAlSi ₃ O ₈	CaSO ₄ ·2H ₂ O

Table 3. The amounts of cations and anions in the water sample of Anarak disposal facility.

ion	(test1)	(test2)	(test3)
pH	6.7	6.8	6.8
Cs ⁺	<0.5 ppm	<0.5 ppm	<0.5 ppm
Ca ²⁺	959.8 ppm	2168 ppm	1200 ppm
K ⁺	26.52 ppm	43.61 ppm	26.19 ppm
Mg ²⁺	480.1 ppm	383.0 ppm	672.2 ppm
Na ⁺	12250 ppm	18396 ppm	13270 ppm
Sr ²⁺	27.01 ppm	u.d.	u.d.
Cl ⁻	2407 ppm	22.5 ppm	16.62 ppm
SO ₄ ²⁻	8400 ppm	8300 ppm	7020 ppm
NO ₃ ⁻	<10	u.d.	0.4

u.d.= undetectable

It is estimated that 30% of the rock volume is composed of montmorillonite clay mineral. This mineral is the main component of bentonite and has high adsorption and swelling. The presence of high amounts of montmorillonite in the rock can be considered in the interpretation of the distribution coefficient of radionuclides. This is because this mineral can have an important role in their distribution coefficient through the adsorption of radionuclides and swelling due to water absorption. In three random samples, the amounts of cations and anions (calcium, magnesium, sodium, potassium, chloride, nitrate, and sulfate) determined by the EPA were determined by ICP and AAS. Table 3 presents the results. According to the result, the groundwater is neutral and weakly saline with low ionic strength and few competing ions for studied ions. Only the concentration of sulfate ions in water is a little high, which may interfere with the adsorption of cations that form insoluble sulfate salts, which does not pose any particular problem for our ions in this experiment.

3.2 Distribution coefficient determination

The values of uranium and thorium distribution coefficients calculated by the batch method for different mean grain sizes of crushed bedrock particles as well as three different concentrations of the desired ion solution are demonstrated in Figure 5 and Table 4. To obtain K_d at intact bedrock, the diagram of K_d vs mean grain size must be matched to the Power equation expressed as follows [16].

$$K_d = a * d^{-b} \quad (3)$$

In this Equation, a and b are the fitting parameters and d is the average particle size in millimeters. To obtain the ion distribution coefficient in unbroken bedrock, it is sufficient to draw a line parallel to the x-axis from the end of the adsorption curve to obtain the y-intercept point that is $K_{d \text{ int}}$. Figure 5 and Table 4 shows K_d changes vs the mean grain size of crushed bedrock samples at different concentrations of thorium and uranium ions. By increasing the

size of particles, K_d decreases and remains almost constant at the end of the diagram for both. The explanation for this observation can be related to the increase in the surface area of particles due to fragmentation, and consequently the increase in the contact surface of ions with the active adsorption sites in the particles and thus K_d . Also, K_d calculated at three studied concentrations shows that the differences in smaller particles are noticeable but can be ignored in larger particles, so that K_d curves for two concentrations of 100 and 10 ppm overlap at the bottom of the chart. Therefore, it can be concluded that for accurate calculation, it is better to use low concentrations to estimate K_d in unbroken bedrock. On the other hand, the concentration of ions also has a significant effect on the value of K_d due to the formation of different species in the solution and adsorption mechanism. Thus, in order to study the adsorption behavior of these two ions in detail, it is better to determine the types of uranium and thorium speciation in aqueous solution. Since the uranium oxidation state is +VI in our experiments environment, we focus on the chemistry of the prevailing forms of this species. The hydrolysis of hexavalent uranium is not strong but easy. Depending on the uranium concentration, the hydrolysis of hexavalent uranium starts at pH around 3 with the formation of mono and dimeric hydrolysis products, UO_2OH^+ and $(UO_2)_2(OH)_2^{2+}$. The more hydrolyzed product, $(UO_2)_3O(OH)^{3+}$ is formed as the pH increases to around 5-6 and when the pH exceeds 6, the predominant species is the

$UO_2(OH)_2 \cdot H_2O$ free-charge complex (Figure 6). Compared to the uranium ion, the solution chemistry of thorium is more straightforward. This is due to its only one oxidation state, +IV, existing in the environment forming the Th^{4+} ion. Thorium has large ionic radius, so the hydrolysis products and complexes are weak. The hydrolysis products are still rather easily formed in basic conditions. In acidic conditions, the Th^{4+} form is prevailing but already at pH around 3 hydrolysis of products start to become predominant forming mono-, di- and tri valent hydroxides as $Th(OH)^{3+}$, $Th(OH)_2^{2+}$ and $Th(OH)_3^+$. In more neutral and basic conditions, above pH 6, the predominant species is $Th(OH)_4$ (aq) in low Th concentrations. In higher concentrations of both ions around 2×10^{-4} M polynuclear species, such as $M_6(OH)_{15}^{9+}$ and colloid formation become of great importance competing with the precipitation of the mononuclear species $M(OH)_4$ or $M(OH)_6$, which is insoluble in aqueous solution [20]. This is why the data calculated for the distribution coefficient are not reliable at a concentration of 1000 ppm of thorium and uranium ions. Given that the predominant species of both ions at concentrations of 10 and 100 ppm and pH around 7 of local- water are free-charge complexes. It can be concluded that the mechanism of adsorption of thorium and uranium ions does not occur through ion exchange process. Therefore, weak chemical interactions or physical adsorption can explain the adsorption mechanism.

Table 4. Data extracted from distribution coefficient change diagrams according to the average size of crushed bedrock particles for thorium and uranium ions.

ion	Concentration (mg/l)	$K_{d \text{ int}}$ (ml/g)	Maximum of adsorption capacity (Q_0) (mg/l)	a (ml/g)	$-b$
U	1000	2.53	183.8	4.91	0.841
	100	5.93	19.4	9.24	0.526
	10	6.52	2.2	9.55	0.609
Th	1000	4.20	198.6	6.23	0.736
	100	10.61	21.8	13.70	0.476
	10	11.72	2.85	17.53	0.416

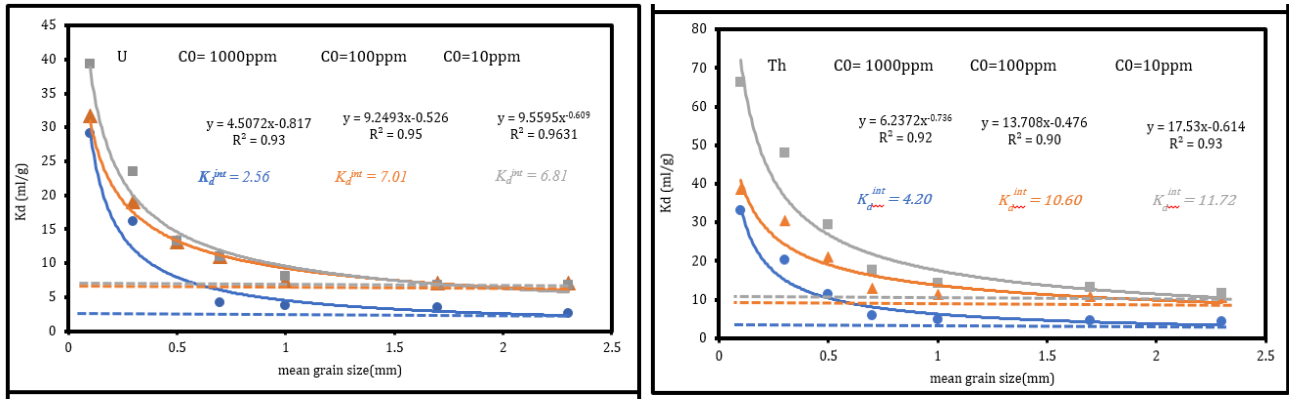


Fig. 5. Distribution coefficient of uranium and thorium versus its primary concentration for different size of crushed bedrock particles.

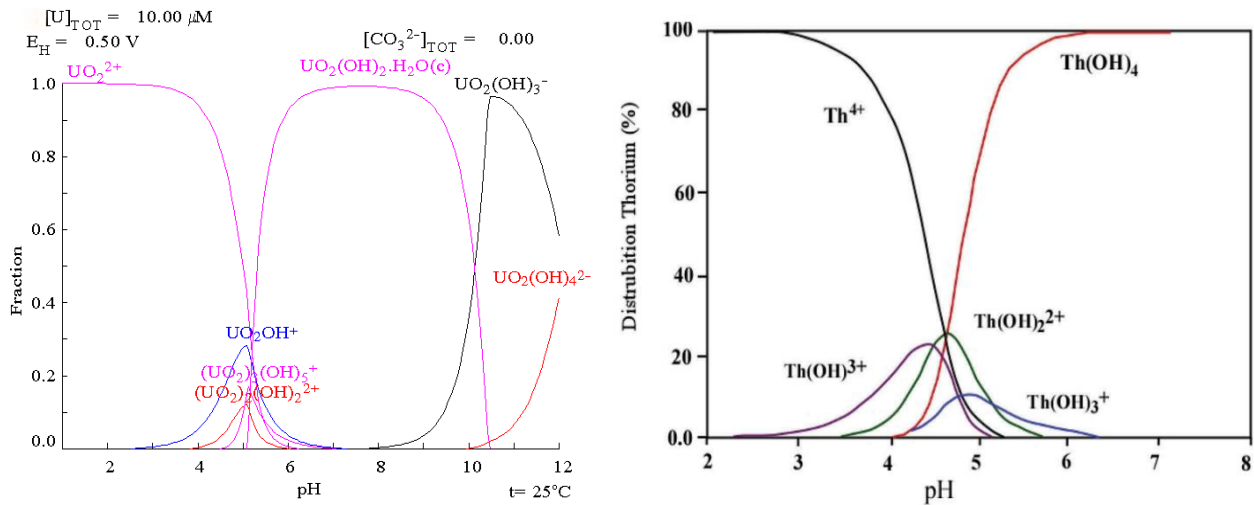


Fig. 6. Eh-pH diagram of low concentration of uranium and thorium in solution (Atlas of Eh-pH diagrams) [20].

4. Conclusion

This research determined the distribution coefficients of uranium and thorium ions in intact bedrock. According to petrography and XRD analysis, the hand specimens are sandstone type that can be classified as "quartz-bearing greywacke" or "quartz-feldspathic greywacke". Among the various known manner sorptions for metal ions and based on the obtained data, our proposed mechanism for sorption of uranium and thorium ions is composed of physical adsorption and chemical interactions. The results of Q_{max} (U (183.8) and Th(196.8) mg/l) extracted from the plot of K_d versus the mean grain size of crushed bedrock particles show that thorium and uranium ions have strong interactions with the bedrock

structure resulting in high adsorption capacity. Also, due to the formation of insoluble complexes of the studied ions at a concentration of 1000 ppm and incorrect calculation of the distribution coefficient, it is better to use initial ion concentrations of 100 and 10 ppm to calculate $K_{d int}$. According to these results, it can be inferred that the bedrock of the Anarak Waste Repository can be a sufficient barrier to prevent infiltration of thorium and uranium ions into the sub-layers. However, other additional barriers may be required with taking into account safety assessment considerations.

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