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Leaching Efficiency of ^{238}U and ^{232}Th series from Granitic Soils

Eficiência de Lixiviação das séries ^{238}U e ^{232}Th de Solos Graníticos

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Abstract

Granitic soil, a uranium (U) and thorium (Th)-bearing soil body, can be sourced to extract fissionable nuclides through leaching using acidic solutions. Limited research indicates the feasibility of extracting U and Th from granitic sources, necessitating further studies to optimize leaching efficiency and its potential as a fissionable fuel. This study investigates the leaching efficiency of the uranium ^{238}U and thorium ^{232}Th series, as well as ^{40}K, in granitic soil samples. The study utilized a high-purity germanium detector (HPGe) to quantify the activity levels of the ^{238}U and ^{232}Th series and ^{40}K in the studied samples. The black sand deposit and tin-tailing samples were also utilized to benchmark the finding of the leaching efficiency of the uranium ^{238}U and thorium ^{232}Th in granitic samples. The study found that the leaching samples contained low radioactivity levels that were a few to tenth times lower compared to the original samples, with tin-tailing samples exhibiting the highest leaching activity for ^{238}U (630.05 Bq l^{-1}) and ^{232}Th (512.17 Bq l^{-1}) and comparable to granitic soil samples ^{238}U (437.7 Bq l^{-1}) and ^{232}Th (23.9 Bq l^{-1}). Sulfuric acid showed the most efficient leaching reagent ~ up to 23% and 32% for ^{232}Th and ^{238}U, respectively. In comparison, nitric and hydrochloric acids exhibited low leaching efficiencies, ~ 1 – 13%. Overall, granitic soil shows comparable leaching rates to conventional sources of natural uranium nuclear fuel, making it a potential secondary and alternative nuclear fuel source.

Keywords: HPGe detector; Radio nuclides; Radioactivity concentration

Resumo

Solo granítico, um corpo de solo contendo urânio (U) e tório (Th), pode ser obtido para extrair nuclídeos fissionáveis por meio de lixiviação usando soluções ácidas. Pesquisas limitadas indicam a viabilidade de extração de U e Th de fontes graníticas, necessitando de mais estudos para otimizar a eficiência de lixiviação e seu potencial como combustível físsil. Este estudo investiga a eficiência de lixiviação das séries de urânio ^{238}U e tório ^{232}Th, bem como do ^{40}K, em amostras de solo granítico. O estudo utilizou um detector de germânio de Alta pureza (HPGe) para quantificar os níveis de atividade das séries ^{238}U e ^{232}Th e ^{40}K nas amostras estudadas. O depósito de areia preta e amostras de rejeitos de estanho também foram utilizados para avaliar a eficiência de lixiviação do urânio ^{238}U e do tório ^{232}Th em amostras graníticas. O estudo descobriu que as amostras de lixiviação continham níveis baixos de radioatividade que algumas eram dez vezes menores em comparação com as amostras originais, com amostras de rejeitos de estanho exibindo a maior atividade de lixiviação para ^{238}U (630.05 Bq l–1) e ^{232}Th (512.17 Bq l^{-1}) e comparável às amostras de solo granítico ^{238}U (437.7 Bq l^{-1}) e ^{232}Th (23.9 Bq l^{-1}). O ácido sulfúrico apresentou o reagente de lixiviação mais eficiente ~ até 23% e 32% para ^{232}Th e ^{238}U, respectivamente. Em comparação, os ácidos nítrico e clorídrico exibiram baixas eficiências de lixiviação, ~ 1 – 13%. No geral, o solo granítico apresenta taxas de lixiviação comparáveis às fontes convencionais de combustível nuclear de urânio natural, tornando-o uma potencial fonte secundária e alternativa de combustível nuclear.

Palavras-chave: Detector HPGe; Radionuclídeos; Concentração de radioatividade

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1 Introduction

Granitic rock is a light-colored, coarse-grained rock consisting of quartz, feldspar, and mica. It is highly durable and often used in construction and monuments due to its high silica content. It is an immense type of igneous which are massive and hard. Granites naturally contain radioactive elements such as uranium (U) and thorium (Th) (Khandaker, Jojo & Kassim 2012).

U is an actinide element in the industry with essential significance in the nuclear reactor fuel for energy production. Naturally, U can exist in oxidizing states IV (+4) and VI (+6). In the tetravalent state (IV), uranium has a +4 charge, while in the hexavalent state (VI), uranium has a +6 charge (Bhargava et al. 2015). The is an element actinide series that exist in a tetravalent oxidation state and has low solvability under all environmental conditions (Edwards & Oliver 2000). The mobility of uranium and thorium is carefully evaluated within the framework of a chemical process, such as employing the phenomenon of leaching using acidic or alkaline solutions, to facilitate the extraction of these elements for nuclear fuel production, thus enabling their indispensable role in the scientific pursuit of sustainable energy generation (Manaa, Negm & El-Magied 2018).

The investigation of U and Th leaching processes for abundant geological materials has remained limited, primarily attributed to the impracticability of conducting comprehensive leaching procedures. The predominant occurrence of uranium (U) and thorium (Th) within intergranular easily soluble phases in granitic formations highlights the potential for substantial leaching of these elements through dilute acid. Nonetheless, the industrial-scale utilization of granite as an ore for extracting uranium and thorium has yet to mature fully, warranting further research and development in this field. A limited investigation undertaken by (Imam, El & Ghanem 2019). exhibits promising indications of the viability and feasibility of extracting uranium (U) and thorium (Th) from granitic sources, presenting a positive outlook for their potential extraction. A contemporary mining technique (Nada, Iman & Ghanem 2007), called "leaching mining" employs a solid-liquid transfer mechanism to facilitate the translocation of valuable constituents from the ore to the leaching solution. The extraction of uranium (U) and thorium (Th) from low-grade materials necessitates comprehensive studies focusing on enhancing leaching efficiency within the leaching process. These investigations are crucial in optimizing the extraction of U and Th from such challenging sources (Mkhatshwa et al. 2020).

Acidic leaching, extensively employed in chemical mineralogy for elemental extraction, surpasses alkaline leaching due to its shorter leaching time, coarse elementary grinding, superior extraction efficiency, and ability to achieve moderate sample concentrations. Various acid types, such as oxalic acid, nitric acid, hydrochloric acid, and sulfuric acid, can be utilized in this process. Sulfuric acid leaching is a conventional analytical method widely applicable for extracting elemental ores and soils, including berafite, uranite, and granite (Huang et al. 2017). This reagent is particularly favored for uranium leaching due to its affordability and accessibility. Furthermore, nitric and hydrochloric acids not only effectively dissolve uranium but also give rise to the formation of undesirable contaminants in the leach solution (Alsaadi et al. 2021), with variant behaviors of ^{238}U series and ^{232}Th series during acidic leaching (Nada, Iman & Ghanem 2007) permitting more leaching efficiency finding.

The main objective of this study is to assess the leaching efficiency of natural radionuclides present in granitic soil, tin tailings, and black sand while also comparing the effectiveness of various leaching reagents utilized in the leaching process. Furthermore, the finding from this work will serve as baseline data for future studies of leaching efficiency.

2 Materials and Methods

2.1 Sample Collection and Preparation

Two samples of granite soil were meticulously collected from Shirbatu Granite Complex, Qasaba (as shown in Figure 1), precisely located at approximately longitude 69°12'2" and latitude 34°35'33" in southeastern Kabul, Afghanistan. Additionally, two reference samples comprising tin tailings and black sand were carefully gathered from the beach of Langkawi, Malaysia. Each bulk sample was initially divided into two containers: one designated for gamma counting and the other for leaching. For gamma counting, each bulk sample, weighing approximately 500 g, underwent a thorough drying process at 110°C in a hot air oven for approximately 7 hours to eliminate any residual moisture and effectively remove stones and pebbles. Subsequently, the desiccated samples were finely crushed and ground to achieve a particle size falling within the range of 250 – 400 μm. These meticulously homogenized samples were then meticulously packed into standardized 500 ml Marinelli beakers, boasting a height of 7 cm and a diameter of 5.5 cm. The hermetically sealed

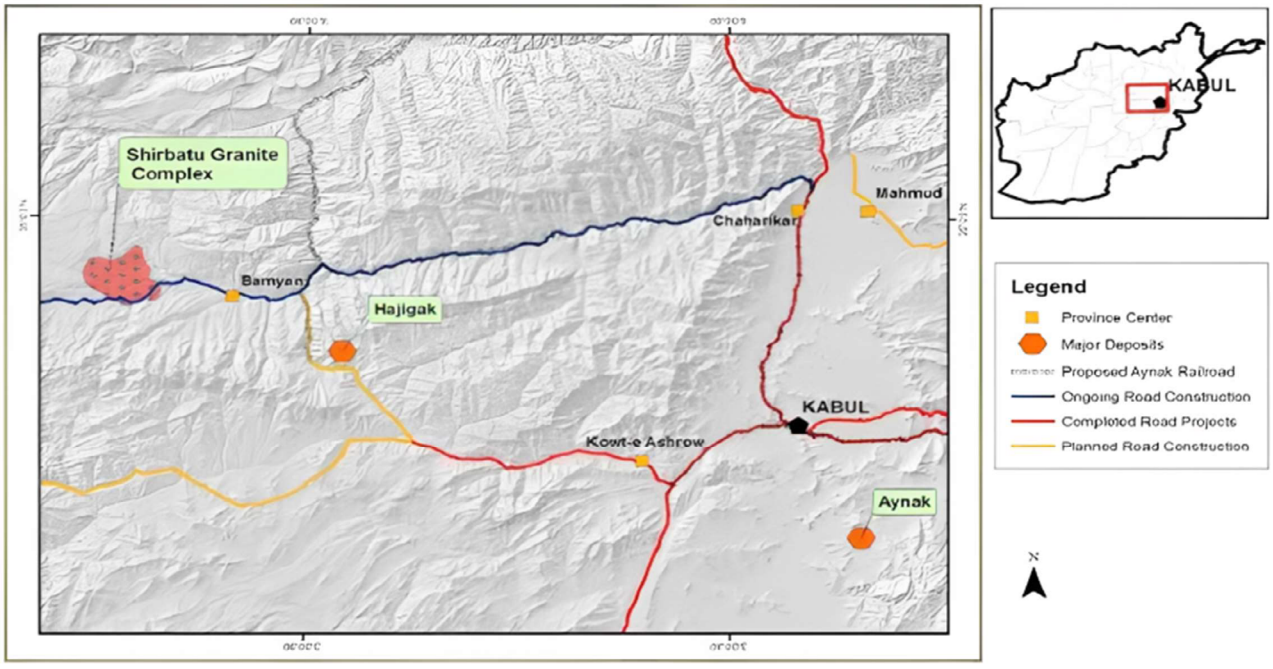

Figure 1 Location map of Shirbatu Granite Complex.

beakers were left undisturbed for one month, establishing secular equilibrium between radium and its decay products. This critical step ensured the confinement of radon gas within the sample volume. At the same time, the decay progeny remained embedded within the sample (Alsaadi et al. 2021). The sealed Marinelli beakers were carefully labeled for accurate sample identification and stored under ambient room temperature conditions until further analysis using an HPGe gamma spectrometer.

2.2 Leaching Procedure

The leaching experiments were conducted with mechanical agitation; four samples (2 granites, one tin tailings, and one black sand sample) were subjected to several agitation leaching experiments using different acid solutions with different concentrations, such as hydrochloric acid (HCL) 37%, nitric acid (HNO3) 65 %, and sulfuric acid (H2SO4) 93 % under the conditions Table 1. After the leaching process, the solutions were rapidly filtered by using filter paper to get pregnant solutions; the filtrate or pregnant liquor of each sample was calibrated to a specific volume of about 500 ml Marinelli beakers and packed well before being checked for ^{238}U, ^{232}Th, and ^{40}K. The beakers were sealed and kept for one month to reach secular equilibrium. Then, the liquid samples were analyzed by gamma spectrometry using HPGe detector to estimate the content of desired nuclides in the leaching solutions. The following Equation 1 was used to determine the leaching efficiency of radionuclides:

Table 1 Conditions for acid pre-leach used in this study.

Type of acid	Concentration (%)	Contact time (h)	Temperature (°C)	Stirring
HCL	37	4 h	27	yes
HNO3	65	4 h	27	yes
H_2SO_4	93	4 h	27	yes

$$\text{Leaching efficiency } \% = \frac{\text{Activity concentration in leachate (Bq)}}{\text{Activity concentration in the original sample (Bq)}} \times 100 \qquad (1)$$

2.3 Analytical Techniques

Gamma spectroscopy was utilized to quantify radionuclide activity level using ORTEC GEM Series P-type coaxial HPGe spectrometer (GEM20-76-LB-C-SMPCFG-SV-LB-76 with 33% relative efficiency and 1.8 keV FWHM resolution and equipped with Mobius cooling system. The instrument allows for high-resolution gamma spectroscopy in the energy range of 40 keV to multiple MeV. The instrument uses high-resolution gamma spectroscopy software (GAMMAVISION 8) for spectral analysis of the gamma emissions. The energy and efficiency calibration of the gamma spectra was conducted using multi-nuclides sources in both powder and water matrix, which are constituted by ^{210}Pb, ^{241}Am, ^{109}Cd, ^{57}Co, ^{123m}Te, ^{51}Cr, ^{113}Sn, ^{85}Sr, ^{137}Cs, ^{88}Y and ^{60}Co. The system was calibrated (as shown in Figure 2) for energy to display gamma photo peaks between 47 and 1836 keV. A period of 21600 s was adjusted for each sample, and the above-invocated software was used for gamma spectrum analysis. The following gamma-ray transitions were used to identify the assigned nuclides' concentrations in the series: ^{238}U and ^{232}Th were indirectly determined from the gamma-rays emitted by their daughter products ^{214}Pb (295.28, 351.97) keV, ^{214}Bi (609.42, 1765.06) keV and ^{212}Pb (238.75) keV, ^{228}Ac (338.37, 911.33) keV, ^{208}TI (510.82, 583.29) keV, ^{212}Bi (727.4) keV respectively. The specific activity concentration of ^{40}K was determined using the 1460 keV of gamma-ray energy.

The activity concentrations of these radionuclides were measured by using the relation given in Equation 72 (Arunima et al. 2021).

$$A\,(i) = \frac{N}{t \cdot Br(\gamma) \times m \times \varepsilon} \qquad (2)$$

is the activity concentration of radionuclide i in Bq kg^{-1}, $Br(\gamma)$ is the emission probability of the gamma line corresponding to the peak energy of radionuclide i, ε is the spectrometer's efficiency corresponding to the peak energy i at the specific geometry, N is the net count under the peak area of the selected gamma line for the measured sample, t is the real counting time, and m is the mass of the sample in kg.

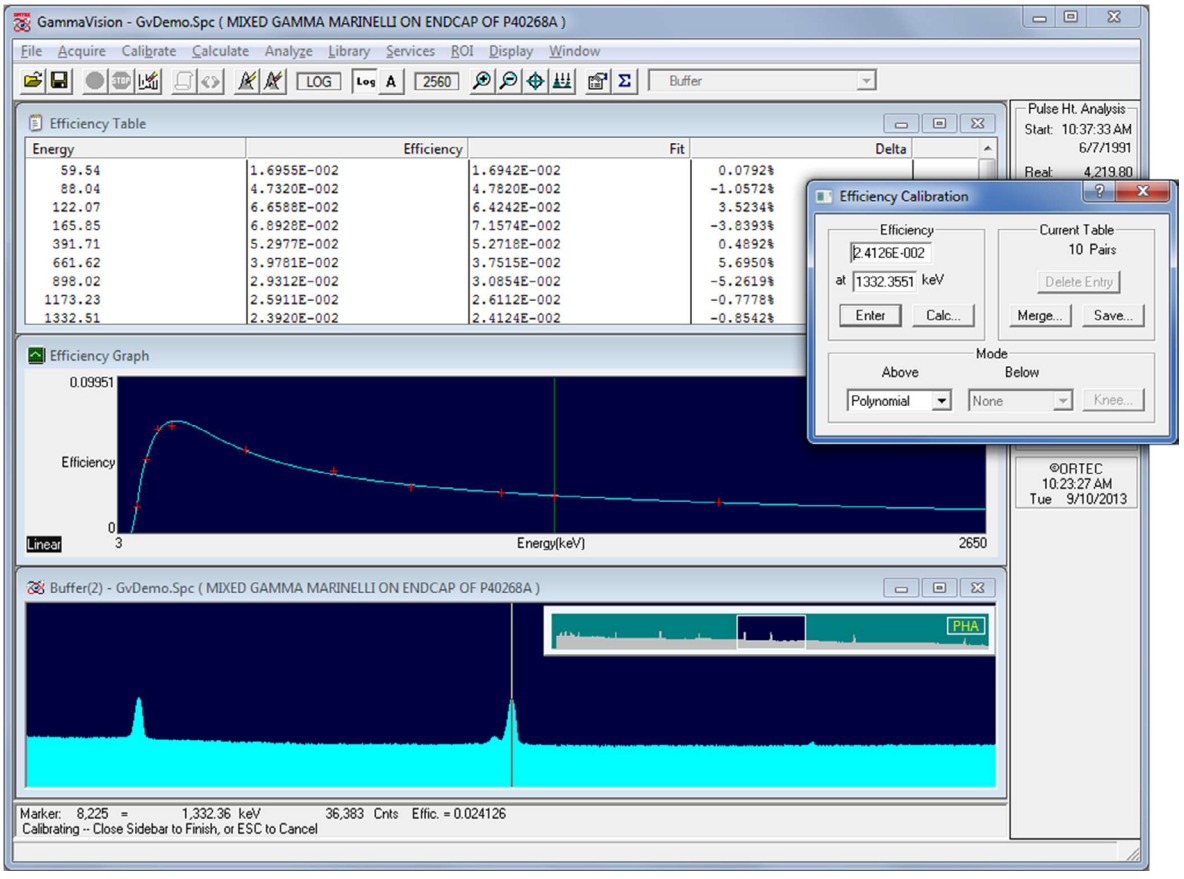

Figure 2 Energy and efficiency calibration of HPGe detector system using GAMMAVISION 8 to quantify radionuclide activity levels in samples.

3 Results and Discussion

Tables 2, 3, 4, and 5 present the gamma activity of radionuclides in both the original samples Bq kg^{-1} and leachates Bq l^{-1}. The ± values for σ variation are not discussed in this study due to the limited sample acquired from this work; therefore, only the calculated activity values are presented in the tables. As the table indicates, nuclide ^{208}TI at 583.29 keV peak energy in the ^{232}Th series shows a prominent gamma spectrum in the analysis.

Tables 2 and 3 show the ^{238}U series, ^{232}Th series, and ^{40}K activities in granitic soil samples. The high activity of Th in granitic sources indicates a high potential for Th utilization as fissionable fuels (via transmuted ^{233}U) as an alternative to natural U in the future. The measured Th activity is few thousand (>5000 Bq kg^{-1}) in granitic soil. In contrast, natural U activity was measured to be a few hundred Bq kg^{-1}. As shown in Table 2, the Th activity in granitic shows a comparable Th and U activity as in tin tailing activity levels (5686.91 and 4565.77 Bq kg^{-1},

Table 2 Activity concentrations of ^{238}U series, ^{232}Th series, and ^{40}K in granite soil sample (1S) and its leachate (pregnant solution G1-a and G1-b).

Sample type		Activity concentration					
		Granite soil sample 1S (Bq kg^{-1})		Solution sample G1-a (Bq l^{-1})		Solution sample G1-b (Bq l^{-1})	
Series	Nuclide	Specific activity	Average activity	Specific activity	Average activity	Specific activity	Average activity
^{232}Th	^{212}Pb	3465.53	6406.84	337.99	437.69	55.11	82.24
	^{228}Ac	3362.20		276.85		49.58	
	^{208}Tl	366.23		30.65		9.23	
	^{208}Tl	24537.29		1726.37		315.98	
	^{212}Bi	4404.72		287.71		56.72	
	^{228}Ac	3922.49		224.27		43.97	
	^{208}Tl	4789.43		179.98		45.08	
^{238}U	^{214}Pb	227.26	252.15	24.59	23.98	7.81	8.29
	^{214}Pb	225.27		24.44		5.79	
	^{214}Bi	246.61		20.86		5.90	
	^{214}Bi	309.47		26.03		13.66	
	^{40}K	629.14	629.14	130.67	130.67	101.40	101.40

Table 3 Activity concentrations of 238U series, 232Th series, and 40K in granite soil sample (2S) and its leachate (pregnant solution G2-a and G2-b).

Sample type		Activity concentration					
		Granite soil sample 2S (Bq kg^{-1})		Solution sample G2-a (Bq l^{-1})		Solution sample G2-b (Bq l^{-1})	
Series	Nuclide	Specific activity	Average activity	Specific activity	Average activity	Specific activity	Average activity
^{232}Th	^{212}Pb	3970.92	7341.17	73.89	96.84	78.44	116.14
	^{228}Ac	3852.52		68.83		71.98	
	^{208}Tl	419.64		10.21		11.15	
	^{208}Tl	28115.64		329.42		448.70	
	^{212}Bi	5047.08		78.63		82.40	
	^{228}Ac	4494.52		59.95		62.32	
	^{208}Tl	5487.88		56.93		57.98	
^{238}U	^{214}Pb	260.40	252.15	6.63	8.28	7.56	9.25
	^{214}Pb	258.13		6.71		7.31	
	^{214}Bi	282.57		7.80		8.97	
	^{214}Bi	354.60		11.97		13.18	
	^{40}K	720.89	629.14	77	77	77.86	77.86

respectively for Th and U). For the black sand sample, the activity of ^{232}Th and ^{238}U was the highest among other samples.

For all types of samples, the radioactivity levels of their leaching samples were ordered a few times lower than the original activity of primary samples, with the black sand sample showing the highest leaching activity of ^{238}U and ^{232}Th series, among other samples attributed to their substantial original activity levels. Table 6 shows the variation of leaching activity for each sample based on different leaching reagents. As shown in Figure 3, among the three reagents used in this work that is sulfuric, nitric, and hydrochloric acids, regardless of activity levels of the original samples, sulfuric acid shows the most efficient leaching reagent with few tenth % of leaching rate for ^{238}U and ^{232}Th series. Nitric acid and hydrochloric acid show their highest leaching efficiency of 13% for ^{238}U in granitic soil samples, whereas for ^{232}Th series is 6%.

Table 4 Activity concentrations of 238U series, 232Th series, and 40K in tin tailing sample (3S) and its leachate (pregnant solution T3-a and T3-b).

Sample type		Activity concentration					
		Granite soil sample 3S (Bq kg^{-1})		Solution sample T3-a (Bq l^{-1})		Solution sample T3-b (Bq l^{-1})	
Series	Nuclide	Specific activity	Average activity	Specific activity	Average activity	Specific activity	Average activity
^{232}Th	^{212}Pb	3682.11	5686.91	56.08	76.35	43.13	60.57
	^{228}Ac	3340.67		55.13		40.98	
	^{208}Tl	513.90		7.52		6.52	
	^{208}Tl	23080.32		284.67		226.49	
	^{212}Bi	3703.56		50.10		40.63	
	^{228}Ac	2910.91		45.40		35.42	
	^{208}Tl	2573.38		35.54		30.79	
^{238}U	^{214}Pb	4922.16	4565.77	13.06	13.34	11.06	11.30
	^{214}Pb	4924.04		15.45		11.22	
	^{214}Bi	4618.94		12.24		10.23	
	^{214}Bi	3794.52		12.61		12.69	
	^{40}K	54.35	54.35	37.58	37.58	35.96	35.96

Table 5 Activity concentrations of 238U series, 232Th series, and 40K in black sand sample (4S) and its leachate (pregnant solution B4-a and B4-b).

Sample type		Activity concentration					
		Granite soil sample 4S (Bq kg^{-1})		Solution sample B4-a (Bq l^{-1})		Solution sample B4-b (Bq l^{-1})	
Series	Nuclide	Specific activity	Average activity	Specific activity	Average activity	Specific activity	Average activity
^{232}Th	^{212}Pb	17558.71	38834.45	327.25	424.10	295.97	394.11
	^{228}Ac	18399.52		294.83		275.39	
	^{208}Tl	2467.02		28.56		26.22	
	^{208}Tl	151480.30		1638.55		1547.99	
	^{212}Bi	27026.12		270.47		240.69	
	^{228}Ac	23855.22		234.46		212.71	
	^{208}Tl	31054.25		174.61		159.79	
^{238}U	^{214}Pb	5474.13	6628.81	30.87	28.91	24.46	21.73
	^{214}Pb	5872.09		31.29		22.93	
	^{214}Bi	6516.92		28.19		21.21	
	^{214}Bi	8652.10		25.31		18.32	
	^{40}K	2201.49	2201.49	47.84	47.84	49.46	49.46

The measurements of leaching efficiency % in the HPGe detector revealed that leachability is at its highest level, with 31.7% for ^{238}U and 22.8% for ^{232}Th in sample 1S-G1-a. In contrast, the lowest was for ^{238}U, only 1.9%, and ^{232}Th is 0.4% in sample 3S, T3-a. As shown in Figure 4, regardless of the type of leaching reagents, the leaching efficiency of ^{238}U and ^{232}Th series for granitic soils indicated a comparable rate as other conventional sources of natural U; mineral tailing and placer deposit of black sand.

The high activity of measured U from the leaching samples, as indicated in Table 6, can be attributed to several reasons. Uranium occurs naturally in U^{+6} and U^{+4}, with the U^{+6} state being more reactive and sensitive to leaching. In contrast, thorium exists solely in the Th^{+4} state, which rarely dissolves in a leaching solution (Ramasamy et al. 2014). In the leaching process of U using sulfuric acid, the extraction of U from the ore involves an ionic exchange mechanism. This mechanism relies on the ability of sulfuric acid to form complex ions and their interaction with the uranium minerals. When sulfuric acid is added to the ore containing uranium minerals, it dissociates into hydrogen ions (H+) and sulfate ions (SO_4^{-2}). The hydrogen ions play a crucial role in the leaching process. They react with the uranium minerals present in the ore, causing the dissolution of uranium into the solution (Derin et al. 2012). The uranium minerals typically consist of uranium oxides or silicates. The acid attracts the mineral lattice and replaces the uranium atoms, forming soluble uranyl sulfate complexes. The most common complex formed is uranyl sulfate (UO_2SO_4), where sulfate ions surround the uranyl ion ($UO2^{+2}$). The resulting solution, containing the uranyl sulfate complexes, can be further processed to recover uranium through solvent extraction or ion exchange, which selectively removes uranium from the solution.

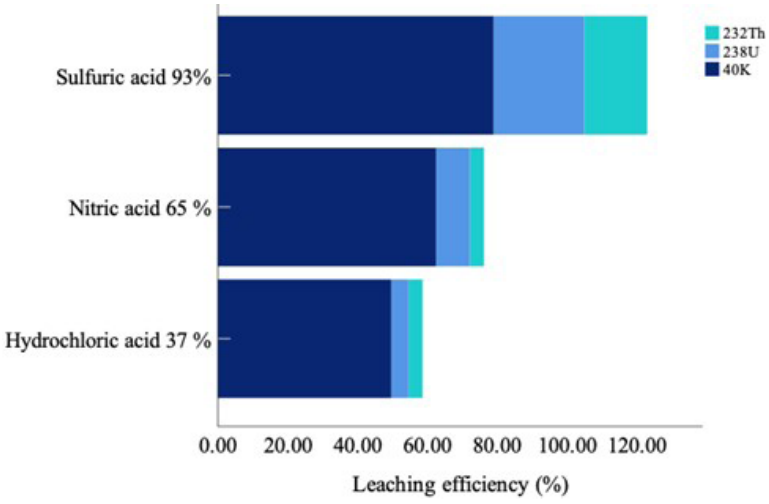

Figure 3 Type of acid against leaching efficiency for different nuclides.

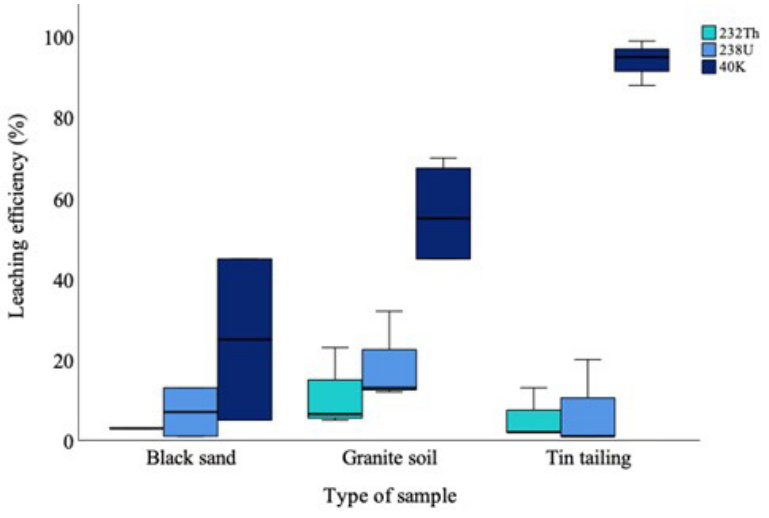

Figure 4 Boxplots of leaching efficiency for different samples.

Table 6 Leaching efficiencies of granite soil sample and other samples using different types of acid.

Sample (label)	Nuclide	Reagent	Original activity Bq kg^{-1}	Leaching activity Bq l^{-1}	Leaching Efficiency (%)
	^{232}Th		1922.05	437.7	23
Granite soil (1S-G1-a)	^{238}U	Sulfuric acid 93%	75.6	23.9	32
	^{40}K		188.7	130.7	70
	^{232}Th		1601.7	82.24	5
Granite soil (1S-G1-b)	^{238}U	Nitric acid 65 %	63.03	8.29	13
	^{40}K		157.28	101.4	65
	^{232}Th		1761.88	96.84	6
Granite soil (2S-G2-a)	^{238}U	Nitric acid 65 %	69.34	8.28	12
	^{40}K		173.01	77	45
	^{232}Th		1761.88	116.14	7
Granite soil (2S-G2-b)	^{238}U	Hydrochloric acid 37 %	69.34	9.25	13
	^{40}K		173.01	77	45
	^{232}Th		3980.04	76.3	2
Tin tailing (3S-T3-a)	^{238}U	Hydrochloric acid 37 %	3195.2	13.3	1
	^{40}K		37.8	37.6	99
	^{232}Th		3980.04	60.6	2
Tin tailing (3S-T3-b)	^{238}U	Nitric acid 65%	3195.2	11.3	1
	^{40}K		37.8	35.9	95
	^{232}Th		3980.04	512.17	13
Tin tailing (3S-T3-c)	^{238}U	Sulfuric acid 93%	3195.2	630.05	20
	^{40}K		37.8	32.95	88
	^{232}Th		15533.8	424.1	3
Black sand (4S-B4-a)	^{238}U	Hydrochloric acid 37 %	2651.5	28.9	1
	^{40}K		880.6	47.8	5
	^{232}Th		15533.8	394.1	3
Black sand (4S-B4-b)	^{238}U	Nitric acid 65%	69.34	9.25	13
	^{40}K		173.01	77.86	45

4 Conclusion

This study found out that the leaching efficiency of ^{238}U and ^{232}Th series for granitic earthy and soil products indicated comparable rates as other conventional sources of natural U; for examples mineral tailing and placer deposit of black sand. Hence, it has high potential to be utilized as secondary sources and alternate for viable nuclear fuel sources in future.

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Author contributions

Ahmad Zaher Ahmadi: writing – review; editing; visualization; investigation; formal analysis; software; resources; data curation. **Mohamad Syazwan Mohd Sanusi**: writing – original draft; methodology; formal analysis; data curation; conceptualization. **Rahmatullah Afghan**: conceptualization; data curation.

Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability statement

The data that support the findings of this study will be available from the corresponding author upon reasonable request and all data included in this study are publicly available in the literature.

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