NEUTRON ACTIVATION ANALYSIS OF URANIUM, THORIUM AND REE DISTRIBUTION PATTERN IN CERTAIN ORES

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Abstract

The nuclear materials of interest such as Uranium and Thorium were determined in certain ore samples (boreholes) collected from different areas in the Kingdom of Saudi Arabia (KSA). Four locations in KSA, namely Jabal Hamra, Jabal Sayid, Jabal Tawlah, and Umm Al-Birak areas were selected and investigated. The Neutron Activation Analysis (NAA) technique and gamma-ray spectrometry (non-destructive technique) were used in this study to determine natural Uranium and Thorium concentrations. In addition, the distribution pattern of the rare earth elements (REE) was determined, as a fingerprint for the ore samples from studied areas. The average value of Uranium concentration in studied areas ranged from 66.4 to 335.5 ppm, the corresponding value for Thorium ranging from 98.0 to 519.7 ppm. The results showed that the average value of Uranium and Thorium concentrations in Jabal Sayid samples were 335.5ppm and 519.7ppm, respectively, which are relatively higher than the corresponding values in other studied areas. Eight rare earth elements were determined in this study (La, Ce, Nd, Sm, Eu, Tb, Lu, and Yb). REE concentrations in samples collected from studied areas ranged as follows: 15.38 - 2766 ppm (Jabal Hamra area), 3.08 - 1518 ppm (Jabal Sayid area), 2.65 - 528.9 ppm (Jabal Tawlah area), and 4.6 - 811.9 ppm (Umm Al-Birak area), respectively. The REE pattern and the concentration of these elements can be used as a signature tool for the ore samples and will contribute to the development of a nuclear forensic database for the studied areas. More attention and further investigation should be concentrated on the area of Jabal Sayid where the highest U and Th concentrations were found.

Keywords: *Uranium, Thorium, Neutron activation analysis, Rare earth elements, Distribution pattern*

Introduction

The determination of any radionuclide may be achieved through three types of analyses, namely: direct activity measurement (a radiometric analysis), mass measurement technique, and neutron activation analysis technique (NAA).

NAA is an useful analytical procedure for the isotopic mass measurements based on a nuclear reaction followed by a radiometric measurement (gamma-ray spectrometry) of the daughter products from those reactions. It is based on the determination of the gamma radiation-induced in a sample after irradiation with neutrons.

In NAA, the sample is subject to irradiation by a neutron flux inside the nuclear reactor or in any nuclear facility and the radioactive isotopes for the elements are generated. The generated isotopes decay to a lower energy state and emit delayed gamma rays or prompt radiation with characteristic energies for each element. Based on the intensities of the energy characterizing the gamma rays emitted from the isotopes it is possible to determine its concentration level. The advantages of using this technique reside in its highly sensitivity, simplicity and ability to analyze simultaneously several multi-elements (more than 30 elements) without destroying the sample.

NAA technique is accurate in determining a ppm level for both Uranium and Thorium in several different matrices, regardless of the chemical or physical form of the analyzed samples.

Before using the Uranium in the reactor as a nuclear fuel its chemical and physical characteristics need to be verified. The condition of a material used in nuclear fuel depends on several factors, the most important one being its purity [1-13]. The trace impurities presence in U-based fuel is an important factor characterizing the nuclear fuel, because it will affect the neutron economy and the metallurgical characteristics of the nuclear fuel. The most effective tool for defining the geologic provenance of the Uranium ore concentrate is REE (rare earth elements) signatures of the ore itself, as long as they tend to fractionate within the next step of the nuclear fuel cycle, such as the stage in which Uranium oxide is obtained. REE pattern is one of the predictive tools to determine the nuclear materials origin, thus they provide information on the origin of Uranium ore without the need for a direct comparison. The method of origin assessment through the REE pattern determination, Lead (Pb) dating of the ore used for production, and age of the ore are very important tools to verify the origin of the Uranium concentrate in that ore [9, 14-15].

This study aims to determine the concentration level of Uranium, Thorium and REE in borehole samples collected from four locations in the Kingdom of Saudi Arabia. The concentration of these elements of potential interest and the rare earth elements pattern will contribute to the establishment of the radioactive map and the nuclear forensic database for the studied areas, respectively.

Materials and Methods

Sample collection, preparation, and irradiation by NAA for determination of actinides

Eighteen rock samples (igneous rocks) were collected from different locations in the Kingdom of Saudi Arabia (KSA) as follows: Jabal Hamra, Jabal Sayid, Jabal Twalah and Umm Al-Birak. The samples were provided by the Directorate General of Mineral Resources at Jeddah in the KSA. For Jabal Tawalah area, the depth of the samples ranges from 25.3 to 71.4 m, while the samples of Jabal Hamra and Umm Al-Birak areas were obtained at the ground level. For Jabal Sayid area, the depth of the samples ranges from 18 to 27 m. Fig. 1 shows the samples locations map for the four studied areas in the KSA.

About 60 mg from each sample was prepared in high purity quartz vials and sealed under vacuum. The samples and the standard were wrapped with Aluminum foil to form bundles, which fit inside the Aluminum irradiation cans. The samples were simultaneously irradiated by a neutron flux of 5×10^{13} neutrons/cm^{2}s, at the University of Missouri Research Reactor (MURR) Center, Colombia, USA. Measurements of U (^{239}Np) and Th (^{233}Pa) in the samples were performed after a cooling time of 6 days and 25.6days, respectively. The sample identification was based on the following nuclear reactions: ^{238}U (n,γ) ^{239}Np and ^{232}Th (n,γ) ^{233}Pa. After irradiation, the samples were analyzed by a gamma-ray spectrometry. Table 1 shows the gamma transitions of Uranium and Thorium.

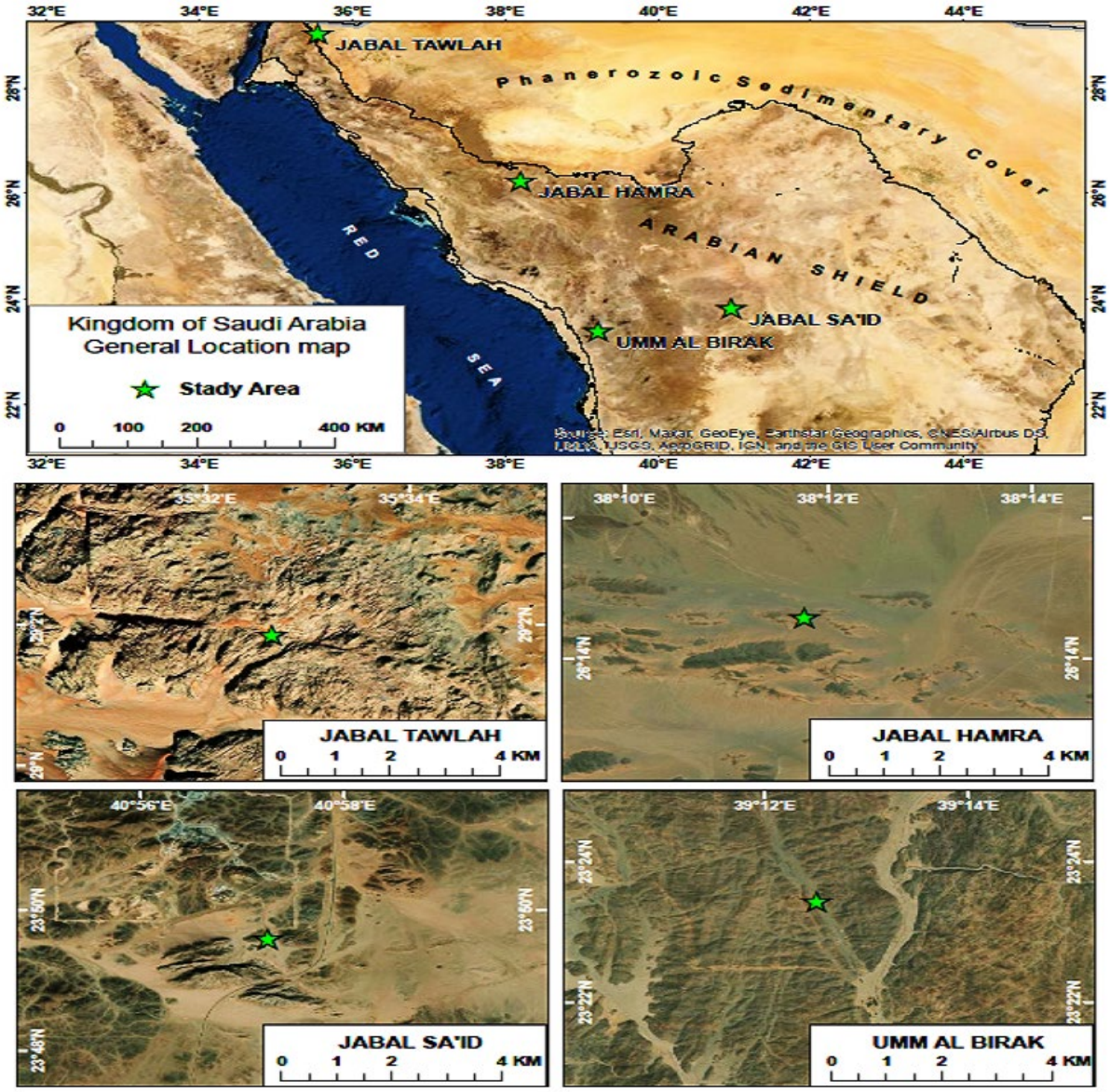


Fig. 1. Map showing the source locations for the studied areas

Table 1. Main gamma transitions produced from ^{238}U and ^{232}Th irradiation

Element	Produced Isotope	Energy (keV)	Intensity (%)
^{238}U	^{239}Np (228.2 keV)	106.12	26.3
		228.2	11.1
		277.6	14.4
^{232}Th	^{233}Pa (312.0 keV)	300.1	6.6
		312.0	38.3
		340.5	4.5

The natural Uranium and Thorium concentrations were determined by measuring the gamma rays from the daughter products of ^{233}Pa and ^{239}Np with associated half-lives and energies of 270 days, 228.2 keV and 2.35 days, 312 keV, respectively.

It is important to mention that the standards and the analyzed samples were simultaneously irradiated and counted in an uniform and consistent way to minimize the effects of neutron flux variation. The standard samples were irradiated at the same conditions for quality control of the calculations and results. The concentrations of ^{238}U and ^{232}Th in the investigated samples are measured in ppm. The $^{232}Th/^{238}U$ ratios are also presented in this study.

Three replicate samples from the National Bureau of Standards (NBS) standard reference material (SRM-1633a, Fly Ash) were used as standards during the analysis of the samples.

The gamma-ray spectrometry used in the analysis had the following specifications: the type of gamma spectrometry is Ge (Li) detector with an efficiency of 18% relative to a "3×3"Na-I detector. The gamma spectrum characterizing each measured sample was obtained by using a Ge (Li) detector type with an energy resolution of 2.2 keV for the 1332.4 keV energy line of ^{60}Co. The gamma emissions from the irradiated samples were measured by using high purity germanium detector (HPGe), which has a resolving power of 1.95 keV at 1332.5 keV energy line of ^{60}Co. The optimum conditions (e.g. measuring time, distance from the detector, and the sample mass) were applied to get the best results. The samples and the standards were irradiated and counted under identical conditions and geometry. The total concentration of Thorium was determined through the detection of ^{233}Pa which was produced by neutron irradiation of the ^{232}Th target nuclide. The concentration of the REE was measured in ppm for about eight elements, as follows: La, Ce, Nd, Sm, Eu, Tb, Lu, and Yb.

Results and discussions

Table 2 shows the results of the Uranium and Thorium concentration measurements, in ppm, for all the investigated samples. $^{232}Th/^{238}U$ ratio (ratio of the isotopes concentration) for the analyzed samples is also given.

Table 2. Uranium and Thorium concentration (ppm) and $^{232}Th/^{238}U$ ratio in the analyzed samples

Sample	*Location*	*$^{238}U(^{239}Np)$ (ppm)*	*$^{232}Th(^{233}Pa)$ (ppm)*	*$^{232}Th/^{238}U$*
1	Jabal Hamra	68.76	264.8	3.85
2	Jabal Hamra	60.7	416.8	6.86
3	Jabal Hamra	109.7	248.1	2.26
4	Jabal Hamra	63.9	131.7	2.06
5	Jabal Hamra	59.7	130.6	2.18
6	Jabal Hamra	73.1	158.8	2.17
7	Jabal Sayid	427.5	743.2	1.74
8	Jabal Sayid	556.2	791.8	1.42
9	Jabal Sayid	22.8	24.0	1.05
10	Jabal Tawlah	212.7	117.9	0.55
11	Jabal Tawlah	95.1	580.6	6.11
12	Jabal Tawlah	112.2	685.6	6.11
13	Umm Al-Birak	104.4	152.8	1.46
14	Umm Al-Birak	109.7	90.0	0.82
15	Umm Al-Birak	52.7	19.7	0.37
16	Umm Al-Birak	53.8	22.4	0.42
17	Umm Al-Birak	31.5	140.2	4.45
18	Umm Al-Birak	46.2	163.0	3.53

The Uranium concentration in Jabal Hamra area samples ranged from 59.7 to 109.7 ppm, with an average value of 72.64 ppm. In the Jabal Sayid area, Uranium concentration ranged from 22.8 to 556.2 ppm, with an average value of 335.5 ppm. The corresponding Uranium concentrations in the samples collected from area of Jabal Tawlah ranged from 95.1 to 212.7 ppm, with an average value of 140 ppm. Uranium concentration for the Umm Al-Birak area samples ranged from 31.5 to 109.7 ppm, with an average value of 66.4 ppm. The highest Uranium concentration (556.2 ppm) was found in Jabal Sayid ore sample.

High concentrations of Thorium were found in all the analyzed samples, with relatively high concentrations up to 792 ppm, except for sample number 9 from Jabal Sayid area and samples number 15 and16 from Umm Al-Birak area, with Thorium concentration of about 20 - 24 ppm. The concentration of Thorium from Jabal Hamra area samples ranged from 130.6 to 416.8 ppm, with an average value of 225.1 ppm. The corresponding values for Jabal Sayid, Jabal Tawlah, and Umm Al-Birak areas ranged from 24.0 to 791.8 ppm, with an average value of 519.7 ppm, 117.9 to 685.6 ppm, with an average value of 461.4 ppm, and 19.7 to 163.0 ppm, with an average value of 98.0 ppm, respectively.

The origin of radioactive contamination can be assessed through the determination of each radionuclide elemental concentration ratios in the sample, and the level of naturally occurring radionuclides such as ^{238}U and ^{232}Th can be established.

Thorium and Uranium in the samples are observed to be weakly correlated (R^2=0.51), as shown in Fig. 2. The reason for such weakly correlation might be an indication of the distortion in the natural concentration of the measured radionuclides in the ore minerals. Higher values of $^{232}Th/^{238}U$ ratios were found in some areas such as Jabal Hamra (sample number 2 – 6.86) and Jabal Tawlah (samples number 11 and 12 – 6.11), which is an indication of the enrichment in ^{232}Th contents.

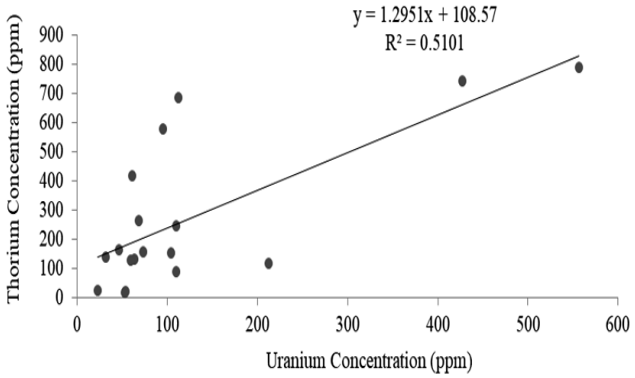


Fig. 2. Correlation between Uranium and Thorium concentration in the analyzed samples

The natural Uranium element is distributed in the Earth's crust, at different levels of concentration in the world, being also found in low-level concentrations in oceans as shown in Table 3. Uranium can be extracted from the mines by either of two techniques (the opencast pits or the underground mining), based on the ore body depth typically up to 250 m deep for the former technique.

From the literature, Uranium is a mobile and potentially bio-available element with a relatively high remobilization potential [16-21]. Naturally occurring ^{232}Th is the raw material from which a new kind of ^{233}U can be created. The interaction of ^{232}Th with neutrons gives first the atom of ^{233}Pa directly in a nuclear reactor, where neutrons are abundant, which then spontaneously transmutes itself into an atom of ^{233}U (an artificial type of Uranium). Then it can be used as a strategic material and doesn't need any enrichment process during its uses.

Table 3. Typical Uranium concentration levels

Concentration levels	**Uranium concentration (ppm)**
Low-grade ore (0.1%)	1,000
Very low grade ore (Namibia) (0.01%)	100
Granite	4-5
Earth's continental crust	2.8
Sedimentary rock	2

The data on Thorium deposits in the world are not yet well documented. Thorium is available as a byproduct of the mining of Uranium and Rare Earth. It has not yet been mined as a strategic primary ore. Table 4 shows that the typical Thorium concentration levels (in ppm) for the Earth crust ranged from 1.2-10.5 ppm. The data obtained from the literature show that Thorium mining is generally easier than the Uranium mining, the advantage of Th mining being that it can be extracted from open-pit monazite deposits. The radioactivity of the mined products is much higher for Th than for U due to the decay chain product of Th such as ^{208}Tl that emits 2.6 MeV gamma rays. However, ^{208}Tl passes into ^{208}Pb (stable) in about 3 min by β decay. The management of Th mine tailings is simpler compared to the U case because of much shorter half-life of one of its daughter products, ^{220}Rd (55 sec), compared to the equivalent daughter product of U, ^{222}Rd (3.8 days).

In the Earth crust, Thorium is geochemically four times more abundant than Uranium. It is difficult to use ^{232}Th in a thermal reactor because it is a fertile isotope, not a fissile one. By absorbing a neutron, Th can transmute to ^{233}U (fissile isotope) and can be easily used in a thermal reactor. In addition, Th has some advantageous physical properties leading to higher conversion rates.

Table 4. Typical Thorium concentration levels

Concentration levels	**Thorium concentration (ppm)**
Average abundance in the Earth's crust	5.6
The average concentration in the upper crust	10.5
The average concentration in the middle crust	6.5
The average concentration in the lower crust	1.2

The ratio of neutron capture to neutron loss (capture) in parasitic material is higher for ^{232}Th than for ^{238}U. Th-based fuel may be utilized either in an open fuel cycle, i.e. by producing ^{233}U in a fuel element and parallel burning it up in the same element, or in a mode with spent fuel reprocessing in a closed fuel cycle [17, 22-26].

Table 5 presents the average for Uranium and Thorium concentration in the analyzed samples collected from the four studied areas. The Uranium concentration level in the Jabal Sayid area of about 336 ppm cannot be considered as a relatively high one. Thorium concentration levels are higher that the Uranium ones in all studied areas.

Table 5. Average Uranium and Thorium concentration levels in the analyzed samples

Region	**Uranium content (ppm)**	**Thorium content (ppm)**
Jabal Hamra	72.64	225.13
Jabal Sayid	335.5	519.7
Jabal Tawlah	140.0	461.4
Umm Al-Birak	66.4	98.0

Fig. 3 shows the average Uranium concentration (in ppm) for the studied areas and their location. The average Thorium concentration (in ppm) for the studied areas and their location is shown in Fig. 4.

Uranium concentrations of 2000 ppm are economically appropriate for the mining process, whereas concentration levels of 1000 ppm should be explored with special technological consideration [27]. Thorium concentration required for the economic mining process is about 60,000 ppm [27]. The registered highest Thorium concentration found in Jabal Sayid area cannot be considered as appropriate for economic mining venture.



Fig. 3. Average Uranium concentration for the studied areas

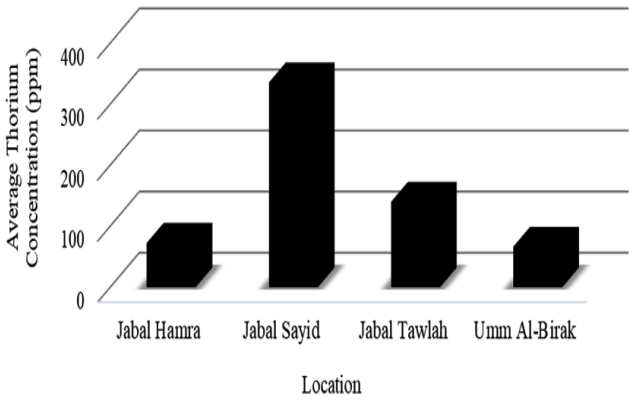


Fig. 4. Average Thorium concentration for the studied areas

In Jabal Sayid area was found the highest content both for Uranium and Thorium, as can be seen in Figs. 3 and 4, and also in Table 5.

In Table 6, the average concentrations of Uranium and Thorium obtained in present study are comparatively presented against similar results from previous published studies in KSA, or carried out abroad. The worldwide average concentration levels are also presented for comparison. The results obtained in present study are consistent with previously published results for areas of interest in KSA.

Table 6. Average Uranium and Thorium concentrations (ppm) for granite rock samples
Comparison between present study and previous published results worldwide

Country	***^{238}U concentration (ppm)***	***^{232}Th concentration (ppm)***	***Reference***
Saudi Arabia (KSA)	230	46.7	[28]
	220	-	[29]
	2396 (1778 - 2256)	3703 (2724 - 4295)	[30]
USA (Western)	12350	81.2	[31]
Italy	892.9	167.8	[28]
Egypt	972.9	180.6	[32]
	1982.7	-	[33]
Sudan	273.1	265.6	[34]
This study	**153.6**	**326**	-
Worldwide	247 - 1482	81 - 326.4	[35]

REE concentrations remains unchanged during the mining and milling process and can thus provide information related to the ore material origin. There are three types of REE, namely: Light REE, Middle REE and Heavy REE, respectively. Light rare elements can be fractionated from heavy rare elements throughout the formation of minerals such as monazite, garnet, apatite, and zircon.

The rare-earth elements concentrations for La, Ce, Nd (as low REE), Sm, Eu, Tb (as middle REE), and Lu and Yb (as high REE) were determined in the analyzed samples collected from the four studied areas and the results are comparatively presented in Table 7, together with the sum of all REE concentrations for each area of study (ΣREE).

The table also contains Eu/Sm and Ce/La concentration ratios which represent a measure indicator for the degree of light REE fraction relative to the high REE.

REEs concentrations in the samples collected from selected areas were found to be in the following ranges: 15.38 - 2766 ppm (Jabal Hamra area), 3.08 - 1518 ppm (Jabal Sayid area), 2.65 - 528.9 ppm (Jabal Tawlah area), and 4.6 - 811.9 ppm (Umm Al-Birak area), respectively.

The lowest registered REE concentrations were recorded for Eu in all collected samples, except for Jabal Hamra area. In the samples from Jabal Hamra area the lowest concentration was obtained for Lu (15.38 ppm), followed by Eu (24.93 ppm). The highest REE concentrations were recorded for Ce (2766 ppm in Jabal Hamra and 811.9 ppm in Umm Al-Birak areas samples), La (1518 ppm in Jabal Sayid area samples), and Yb (528.9 ppm in Jabal Tawlah area samples), respectively.

The ΣREE values increased from 1278 ppm (the lowest value) in Jabal Tawlah area samples, to 1605 ppm in Umm Al-Birak area samples, 2292 ppm in Jabal Sayid area samples, and finally to 5493 ppm (the highest value) in Jabal Hamra area samples, respectively.

REE distribution pattern for the analyzed samples is illustrated in Fig. 5.

Table 7. Rare earth elements concentrations in the analyzed samples (ppm)

Elements / Ratios	***Concentration (ppm)***			
	Jabal Hamra	***Jabal Sayid***	***Jabal Tawlah***	***Umm Al-Birak***
La	1090.00	1518.00	71.89	316.10
Ce	2766.00	405.50	304.80	811.90
Nd	1206.00	251.90	175.30	324.50
Sm	226.00	66.86	71.01	81.27
Eu	24.93	3.08	2.65	4.96
Tb	38.85	11.55	50.75	13.35
Lu	15.38	3.83	73.09	4.60
Yb	126.00	31.30	528.90	48.44
ΣREE	5493.00	2292.00	1278.00	1605.00
Eu/Sm	0.11	0.05	0.04	0.06
Ce/La	2.54	0.267	4.24	2.57

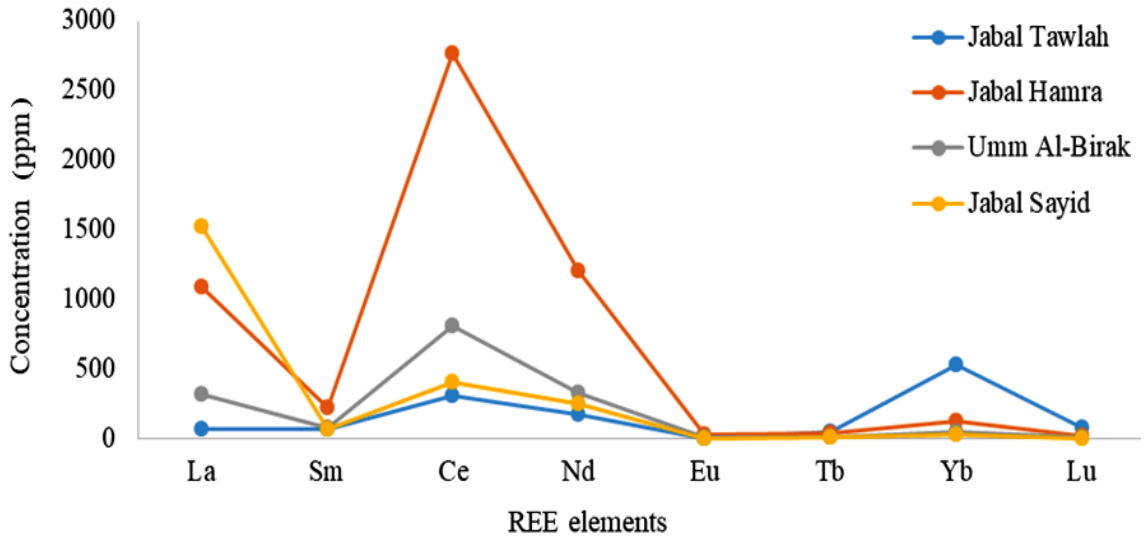


Fig. 5. Rare Earth Elements pattern in the analyzed samples

Conclusions

Uranium, Thorium and REE concentration have been determined by using the Neutron Activation Analysis and Gamma-ray Spectrometry techniques in igneous rocks collected from Jabal Hamra, Jabal Sayid, Jabal Tawlah and Umm Al-Birak areas in KSA.

The results revealed that the samples collected from Jabal Sayid area had the highest value for Uranium and Thorium concentrations, the average content of U and Th being of 336 ppm and 520 ppm, respectively. The lowest average content of U and Th was obtained in the samples collected from Um-Al-Birak area, with values of 66 ppm for U and 98 ppm for Th, respectively.

Despite the fact that the U and Th concentration levels in Jabal Sayid area samples were relatively high, according to economic considerations [27] cannot sustain launching of any mining activities.

As for the $^{238}U/^{232}Th$ ratio, the highest values obtained were 6.86 (sample no 2 from Jabal Hamra area) and 6.11 (samples no 11 and 12 from Jabal Tawlah area), respectively.

The rare earth elements were found in association with Thorium in the collected samples. The highest value of ΣREE concentration (sum of all analyzed REE - La, Ce, Nd, Sm, Eu, Tb, Yb, Lu) was obtained for the samples collected from Jabal Hamra area (5493 ppm), the lowest value of ΣREE concentration being obtained for the Jabal Tawlah area samples (1278 ppm).

The rare-earth patterns were also determined for the studied areas and may be used in the future as a signature tool for the assessment of the fingerprint of the Uranium contents in considered areas.

Taking into consideration that the Jabal Sayid area was characterized by the highest Uranium and Thorium concentrations, more investigation and analyses need to be performed by collecting a high number of replicates samples at various depths.

Moreover, the sample analysis could be also performed by using an alternative analytical method, such as the Inductively Coupled Plasma Mass Spectrometry technique. The advantage of this alternative technique is given by its higher detection limit, good precision, short time of analysis and the possibility to analyze a large number of samples with low-cost analysis.

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