

RARE EARTH ELEMENTS IN EGYPTIAN GRANITE BY INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS

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The mobilization of rare earth elements (REEs) in the environment requires monitoring of these elements in environmental matrices, in which they are mainly present at trace levels. The similarity in (REEs) chemical behavior makes the separate determination of each element by chemical methods difficult; instrumental neutron activation analysis (INAA), based on nuclear properties of the elements to be determined, is a method of choice in trace analysis of (REEs) and related elements. Therefore, (INAA) was applied as a sensitive nondestructive analytical tool for the determination of rare earth elements to find out what information could be obtained about the (REEs) of some Egyptian granite collected from four locations in Aswan area in south Egypt as follows wadi El-Allaqi, El-Shelal, Gabel Ibrahim Pacha and from Sehyel Island and to estimate the accuracy, reproducibility and detection limit of NAA method in case of the given samples. The samples were properly prepared together with standards and simultaneously irradiated in a neutron flux of 7×10^{11} n/cm².s in the TRIGA Mainz research reactor facilities. The following elements have been determined: La, Ce, Nd, Sm, Eu, Yb and Lu. The gamma spectra was collected by HPGe detector and the analysis was done by means of computerized multichannel analyzer. The X-ray fluorescence XRF was also used.

Key words: *Rare Earth Elements/ Neutron Activation Analysis/ XRF*

INTRODUCTION

The rare earth elements (REEs) are a group of elements with very similar chemical characteristics. This is due to their similar ionic radii and electronic configuration ⁽¹⁾. REEs play an important role in geochemical studies, since their distribution in earth crust and mantle contributes to elucidate evolutionary processes of geological cycles, providing information on derivation and dating of igneous rocks ⁽²⁻³⁾. REEs are becoming more and more technologically significant due to their widespread utility as fine chemicals in modern industry (metallurgy, oil refinery, glass, ceramics, electronics, nuclear engineering) as well as in high-tech applications semi-conductors, super-conductors, optics and laser researches ⁽⁴⁻⁵⁾. In clinical diagnostics, complexes of lanthanide ions with encapsulating ligands are proposed as luminescent labels in fluoroimmunoassays ⁽⁶⁾. The demand of (REEs) for industrial applications is increasing rapidly, and concurrently the environmental contamination by (REEs) in biological samples as plants and human organs may increase, requiring monitoring of these elements both in environmental and in biological matrices, in which they are mainly present at trace levels ⁽⁷⁾. Investigations on (REEs) in the environment depend on sensitive analytical techniques, due to the low concentration values of these elements. Even modern analytical techniques like ICP/MS may require a

preconcentration step, especially for elements like Lu, which often occurs in ng/kg levels. To assure the reliability of the data, a critical verification of analytical values must be performed and quality control must be of the order of the days. The accuracy of the analytical results is normally verified by application of analytical procedure to certified reference materials. Nuclear analytical techniques with their broad band of applicability to almost all matrix types and their exceptional sensitivity to many elements, are an indispensable tool for environmental research. Neutron activation analysis (NAA) appears to be an attractive technique for determining heavy metals and REEs present in environmental samples; soil, sediments, plants, water, air particulates, etc, ⁽⁸⁾. NAA has been applied to cosmochemical and geochemical problems from the time the first nuclear research reactors 1940s. Undoubtedly the most significant contribution of early NAA to modern geo- and cosmochemistry was the work dealing with the distribution of the (REEs) in meteorites, rocks and sediments. NAA is frequently used to obtain data for geological materials. Usually this technique provides good results within a reasonable timescale. The accuracy and the limit of detection of the (REEs) data using INAA depends, however, strongly on the type of material analysed and on the content of several interfering elements which provide either a high background or overall activity and therefore may prohibit reliable (REEs) analysis. One major difficulty in the neutron activation analysis of rare earth concentrations lies in the high absorption cross section of several isotopes. Table (1) shows the potentials of the different methods ⁽⁹⁻¹⁰⁾. However, it should be noted that the numerical values refer mostly to the ideal case (i.e. the absence of any interfering element and assuming optimum experimental conditions). Essentially, they can be used only for comparison of the different methods. In practice, one has to consider such factors as the nature of the matrix, interference from simultaneously present (REEs), problems of the pretreatment and incidental chemical separations, time consumption, the achievable accuracy, the given conditions and the cost factors arising from all this. Nevertheless, activation analysis may be the most frequent choice for the solution of analytical problems, as it is the case for the (REEs).

Table 1. Detection limits of REE in nanograms by various techniques ⁽⁹⁻¹⁰⁾.

Elements	X-ray fluorescence	Flame spectrophotometry	Neutron activation	Spark source mass spectroscopy	Emission Spectrography, Cu, spark
La	---	20	0.1	0.1	5
Ce	---	200	0.3	0.1	40
Nd	60	10	10	0.4	30
Sm	60	10	0.05	0.5	40
Eu	30	1	0.0005	0.2	2
Yb	5000	2	0.1	0.5	5
Lu	----	6	0.005	0.1	100

EXPERIMENTAL PROCEDURE

1-Samples preparation and irradiation

The granite rocks in Egypt are broadly classified into main groups old granite and young granite ⁽¹¹⁾. The samples under consideration are classified as follows:

(1) Samples collected from wadi El-Allaqi and Gabel Ibrahim Pacha represent old granite⁽¹²⁻¹³⁾.

(2) Samples collected from El Shalal and Sehyel Island represented young granite⁽¹⁴⁾.

The old granites are characterized by an increase in the plagioclase content over the potash feldspars. The old granite are medium to coarse grained of grey colours composed of plagioclase, quartz and potash feldspars. The young granite has a pink to red colour medium to coarse grain size and it is mainly composed of potash feldspars, quartz and intermediate to sodic plagioclase. Mafic minerals are mostly biotite and to a less extent muscovite. Iron oxides occur as spots of hematite and limonite. Twenty represented granite rock samples collected from four locations in Aswan area as follows, wadi El-Allaqi, El-Shalal, Gabel Ibrahim Pacha and Sehyel Island for investigation by instrumental neutron activation analysis. Table 2. shows the characteristic of the collected samples.

Table 2. Characteristic of the collected samples

Location	Type of rock	Sample name	Age Million years
Wadi El-Allaqi	Igneous	Old Granite	830-927
Gabel Ibrahim Pacha	Igneous	Old Granite	830-927
El-Shalal	Igneous	Young Granite	570-600
Sehyel Island	Igneous	Young Granite	570-600

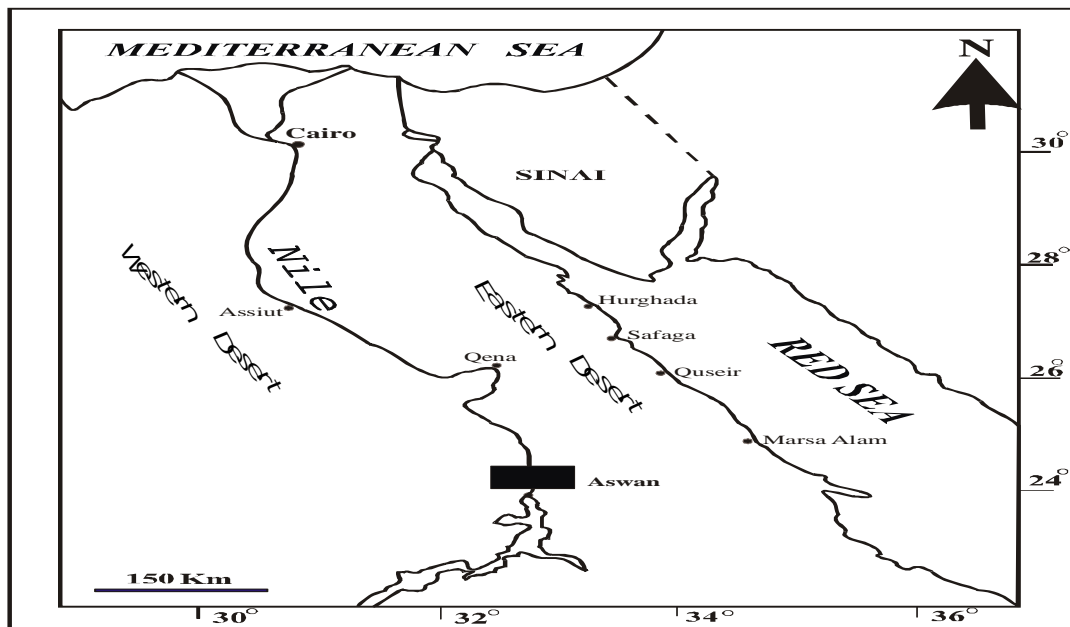


Figure 1. A map showing the location of the studied areas.

Figure 1. shows the location of the studied areas and Figure 2. shows the locations of the granite samples. NAA achieves a qualitative and quantitative analysis of the unknown samples by irradiating them with neutrons (n, γ) reaction and detecting the emitted γ rays from the outgoing radioactive nuclides after irradiation. Qualitative analysis can be achieved by perfect analysis of γ lines in γ spectrum detected and registered by (HPGe) detector and its associated electronic circuit⁽¹⁵⁻¹⁷⁾. The samples have been prepared into finely ground homogenous matered. They were crushed to a diameter range of less than 125 μm and greater than 63 μm . The crushed samples were dried at 105 °C to constant weight.

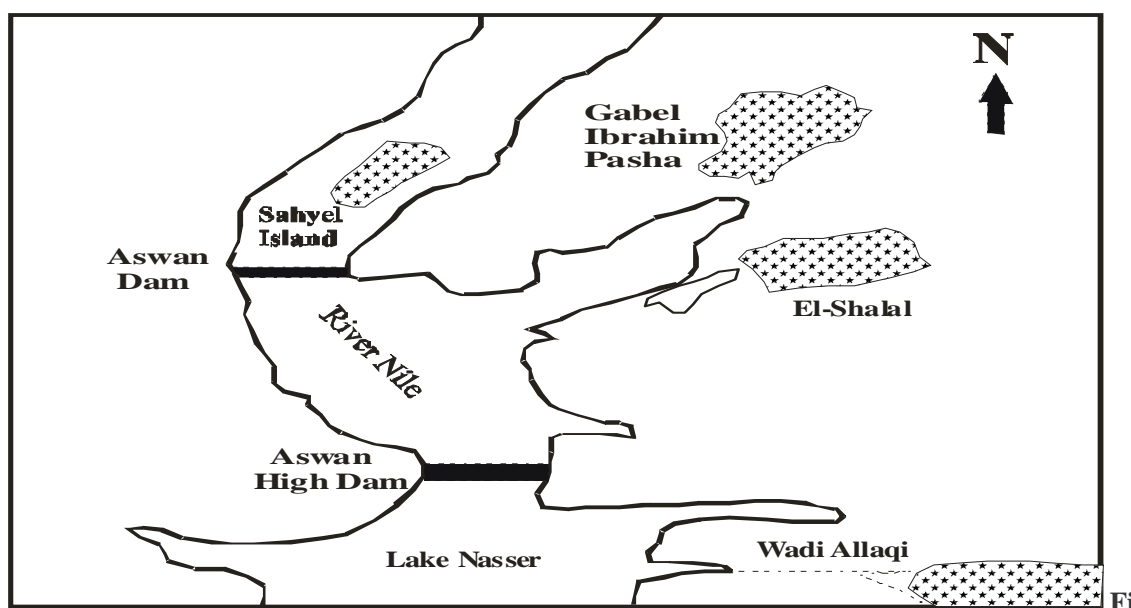


Figure 2. Locations of the granite samples

Polyethylene capsules filled with 100 mg of powder samples were then irradiated with the standard reference material with thermal neutrons at the university of Mainz Triga research reactor (100 KWth) for 6 hours with a flux of $7 \times 10^{11} \text{ n/cm}^2 \cdot \text{s}$. The rare earth element concentrations in our samples were quantitatively determined by comparison with activities of Dolerite WSE reference material which were activated simultaneously. The data were collected for various measurement after appropriate cooling times. The elements La and Sm measured after cooling time 2 days and 1hour measuring time, the elements Ce, Nd, Eu, Yb and Lu measured after cooling time 14 days and measuring time 8 hours⁽¹⁸⁾. Table (3) shows the radioisotopes used to calculate the concentration of the analyzed elements as well as their nuclear data.

Table 3. Activation and measurement conditions for detected element⁽¹⁹⁻²⁰⁾.

Element	Activation product	Energy keV	Abundance intensity %	T1/2	Detection Limits ($\mu\text{g/g}$)
La	¹⁴⁰ La	1596. 2	97	40.3h	0.2
Ce	¹⁴¹ Ce	145.5	49	32.5d	0.3
Nd	¹⁴⁷ Nd	531	13	11d	3.8
Sm	¹⁵³ Sm	103. 23	27	46.3h	0.03

Eu	¹⁵² Eu	1408.11	22.9	13.3y	0.09
Yb	¹⁶⁹ Yb	197.8	34.9	32d	0.05
Lu	¹⁷⁷ Lu	208.36	7.5	161d	0.01

XRF is one of the most important techniques for the analysis of metals and trace elements, which is independent of the chemical form of the element, as INAA. X-rays emitted from an ionized atom have energies characteristic of the element involved; and the intensity of an X-ray is proportional to the concentration of an element and the strength of the ionizing source. Thus, X-ray fluorescence XRF analysis is based on the generation of characteristic X-rays from a sample irradiated by an energetic beam and hence, capable of measuring the concentrations of different elements in the sample. The general advantages of XRF are (a) the extreme multi-element character for almost all elements with $Z > 10$ to 16, its (b) high selectivity, (c) pure instrumental and non-destructive character, (d) much smaller number of spectral lines compared with atomic emission methods, therefore (e) less spectral interferences with respect to wave length and qualitative element identification, follows the sequence of the periodic system (h) most suitable for element contents from 100 % to ca. 5 to 10 ppm, (i) good precisions of 0.1 to 1% for trace levels ca. $> 5 \pm 5\%$, (j) good speed, (k) ease of operation and (l) economy, (m) automated analysis of large numbers of similar samples with help of sample changer, (n) almost independent from the molecular form of the element. For X-ray fluorescence, 8 g₂ gram from the powdered sample and 1.6 gram from wax material are pressed under 300 N/cm² pressure to give us a discs with 4cm diameter. These discs are then measured by XRF⁽⁹⁾.

2-Instrumentation

The applied gamma-ray spectrometer consists of a HPGe detector with its electronic circuit. The detector has the following specifications: energy resolution (FWHM) at 1.33 MeV Co60 is 1.70 keV Peak to Compton ratio Co-60 is 65.2, relative efficiency at 1.33 MeV Co60 is 29.2 %, energy resolution (FWHM) at 1.22 MeV Co-57 is 686 eV, Operation bias voltage is +2000 dc. The detector is connected to the following components : preamplifier, amplifier, ADC converter and MCA. The measurements were performed and analyzed using the Intergamma Software produced by Intertechnique Deutschland GmbH, Mainz, Germany. The electronic dead time in all measurements was less than 10 % and was automatically corrected by the Intergamma software.

Interferences

In neutron activation analysis (NAA), the following three types of interfering nuclear reactions should be taken into account⁽²¹⁾: 1) Nuclides produced by (n,p) and (n, α) reactions on the other heavier elements are often same as the nuclide produced by the (n, γ) reactions. 2) Fission products of uranium are the same as the radionuclides for the determination of some rare earth elements (REEs), zirconium, ruthenium and molybdenum 3) Daughter nuclides which succeed the (n, γ) reactions of different target elements become the same as radionuclides of interest. The isotopes ¹⁴⁰La, ¹⁴¹Ce and ¹⁴⁷Nd commonly used in the activation analysis of the corresponding elements, are also produced by fission of ²³⁵U. For each of these radioisotopes an interference factor, defined as the activity produced by irradiating 1 μg of pure natural uranium divided by the activity produced by irradiating 1 μg of the chosen element, was determined. The fission product correction factor have been determined experimentally by analysis of natural uranium standards using the same conditions as those of granite rock samples. The spectral

interferences with significant influence on the REE analysis data those which are impossible to avoid by choice of a more convenient analytical peak, counting scheme, or peak integration method.

RESULTS & DISCUSSION

With the introduction of high-resolution Ge(Li) γ -ray detectors many trace elements, including the REE, could be determined simultaneously without any chemical separations in a wide variety of rocks and minerals by INAA. INAA became a standard technique for trace element analysis of silicates (low neutron activation cross section, short half-lives and/or low γ -ray abundances in the nuclear decay). Preliminary studies were made to determine optimum conditions to obtain interference free photo-peaks of the desired elements. Irradiation, cooling and counting time were optimized depending on the half-lives of elements analyzed, in order to improve the ratio between the relative activity of each element and the bulk activity of the matrix. The irradiation time for all samples was 6 hours. It is observed that La and Sm can be measured after cooling time 2 days and 1hour measuring time whereas the other REE require cooling time 14 days and measuring time 8 hours. The selection of photopeaks for the analysis is briefly discussed below for each element. For Lanthanum, the high abundance photopeak of ^{140}La at 1596 keV was used, which is free of interference. The other peak at 487 keV cannot be used due to interferences from ^{47}Ca 489 keV and ^{192}Ir 488 keV. For Cerium, the photopeak of ^{140}Ce at 145 keV was used. For Neodymium, the photoppeak of ^{147}Nd at 531 keV which is free of interference was used. The high abundance peak at 91 keV is surrounded by other peaks, which cannot be resolved from the peak of $^{177\text{m}}\text{Lu}$ 113 keV. For Samarium, the isotope ^{153}Sm is used. As far as Europium is concerned, ^{152}Eu has a number of photopeaks where the high abundance peaks at 1408 and 799 keV are free from interference. Both peaks were used for the determination of this element. Another peak at 122 keV cannot be resolved from the 124 keV line of ^{154}Eu . However, the combined peaks can be used as these are obtained from two isotopes of the same element and have similar half-lives. Ytterbium can be determined using the 198 keV peak of ^{169}Yb . The 396 keV peak cannot be resolved from nearby peaks of ^{152}Eu 383 keV and ^{233}Pa 381 keV.

Table 4. Interferences due to energy and reactions

Radio-nuclie	E _γ , keV	Energy interferences			Reaction interferences	
		Interfering isotopes	Interfering energy	Reference energy	Interfering reaction	Interfering contribution μg/ μgu
La ¹⁴⁰	1596.2				U (n,f) ¹⁴⁰ La	0.032
¹⁴¹ Ce	145.5				U (n,f) ¹⁴¹ Ce	0.201
Nd ¹⁴⁷	91.1				U (n,f) ¹⁴⁷ Nd	0.187
		Np ²³⁹	103.23	106.1		
		Yb ¹⁶⁹	207.7	110.9		

For Lutetium, the high abundance peak at 208 keV of ^{177m} Lu was used. In uranium containing samples, this peak cannot be fully resolved from the 210 keV peak of ²³⁹ Np. The interference factors for the elements La, Ce and Nd are given in table (4). The spectral interferences with significant influence on the REE analysis data those which are impossible to avoid by choice of a more convenient analytical peak, counting scheme, or peak integration method. The interferences given in table (4) were corrected for by the corresponding reference peak of interfering isotopes. The average amount of uranium in the old granite samples is (5.18 ± 0.072) ppm, while the corresponding value in young granite is (16.64 ± 2.13) ppm. Table (5) shows the average concentration of the rare earth elements in the granite samples from the four location in ppm

Table 5. The average concentrations of rare earth elements of granites in ppm determined by INAA

Element	Activation product	Energy keV	T _{1/2}	Wadi. Allaqi	G.Ibrahim Pacha	El-Shelal	Syhail Island
Ce	¹⁴¹ Ce	145	32.5d	138 ± 3.4	58 ±1.7	48 ±2.4	21±1.5
Eu	¹⁵² Eu	1408	13.3y	3.2 ±0.1	1.9 ±0.05	0.7 ±0.02	0.2 ±0.01
La	¹⁴⁰ La	1596	40.3h	95.6 ±2.1	87.1 ±2.5	12.5±0.4	4.4 ±0.2
Lu	¹⁷⁷ Lu	208.4	161d	1.0 ±0.03	0.6 ±0.02	0.6 ±0.01	0.6 ±0.02
Nd	¹⁴⁷ Nd	531	11d	73.4 ±5.2	70.4 ±5.9	28.5±2.4	10.9 ±1.05
Sm	¹⁵³ Sm	103	46.3h	16.1 ±0.3	14.7 ±0.4	4.1 ±0.1	1.2 ±0.05
Yb	¹⁶⁹ Yb	198	32d	7.7 ±0.3	5.8 ±0.2	9.7 ± 0.4	4.9 ± 0.3

From the calculated values presented in table 5 the following observation can be reported; The mean values of all elements in the old granite (wadi El Allaqi and Gebel Ibrahim pacha) are higher than the same elements in the young granite (El Shalal and Sehyel Island). Taking into account the obtained results of the different areas under investigation and the factors affecting the presence of REEs. The high concentrations observed in wadi El-Allaqi and Gebel Ibraheem pacha than other areas can be attributed to the following factors: a) The presence of radioactive accessory minerals such as uranothorite b) The granitic rocks contain accessory minerals such as Zircon, Sphene and also monazite and appetite which readily accumulate in limited uranium and thorium ⁽²²⁾. Figure 3. shows also comparison between the concentration of the rare earth elements in the studied areas For XRF we measure one sample from each location. The reproduceability of major elements was < 0.5, for higher concentrations reproduceability are ~ 10 % (2σ). Table (6) shows approximate chemical composition of Granite samples, determined by XRF.

From the table one can see that the concentrations of Al_2O_3 , CaO , Fe_2O_3 , MgO , P_2O_5 and SiO_2 in the old granite greater than that in young granite where the concentration of K_2O and Na_2O in young granite greater than that in old granite. From the geometrical point of view, the REE are “dispersed” (not so rare) elements i.e., they are spread among many common materials, rather than concentrated into a select few. They are “lithophile” i.e., when allowed to distribute themselves among overwhelmingly enter the silicate. However, during igneous processes the REE trend to remain in the porate them. The minerals that do take up REE in their structures will generally show a preference for either light or heavy REE depending on nature of the ionic positions available for substitution. This is a consequence of the regular decrease in ionic radii for the trivalent REE from La to Lu (the so-called lanthanide contraction. When comparing the REE contents of different rocks or minerals, one generally normalizes the concentrations of the individual REE to their abundance in chondrites (the meteorites that best represent the composition of the non-volatile fraction of solar system material). This is done to take into account the fact that the elements with even atomic numbers are more abundant than their neighbours with odd atomic numbers (Oddo-Harkins effect), a consequence of unclear stability. The normalization results in REE patterns (normalized concentration versus atomic number) that are smooth, except for occasional anomalies for Eu and Ce (because of their sometimes differing oxidation states) and for Eu and Yb (the two most volatile REE that are sometimes depleted in the refractory Ca-Al-rich) inclusions present in carbonaceous chondrites).

Table 6. Approximate chemical composition of Granite samples, determined by XRF.

Composition (ppm)	Wadi.Allaqi	Gebel .Ibrahim Pacha	El-Shelal	Syhail Island
Al_2O_3	96500	93000	91500	11100
CaO	40500	14500	6500	7500
Fe_2O_3	110000	30500	11000	12000
K_2O	46850	29400	46550	50050
MgO	11500	3000	500	500
MnO	1510	430	2450	760
Na_2O	14500	1500	13500	20500
P_2O_5	7250	1400	550	700
SiO_2	594000	462000	551500	585500
TiO_2	17800	4150	1800	1200
F	1500	1000	1000	1000
S	130	90	795	350
Ba	1053	711	142	253
Cl	74	11.5	14.5	13
Co	42	4	---	---
Cr	91	49	44	38.5
Cu	19	14.5	13.5	14.5
Mo	3.5	3.5	2.5	1.5
Ni	9	7	6.5	8
Pb	12.5	11.5	16.5	19.5
Se	2	---	1	---
Sn	---	---	1	9

Sr	352	132	34	37
Ti	0.1	0.2	0.55	0.55
V	139	22	7	14.5
Zn	128	47	23.5	30.5

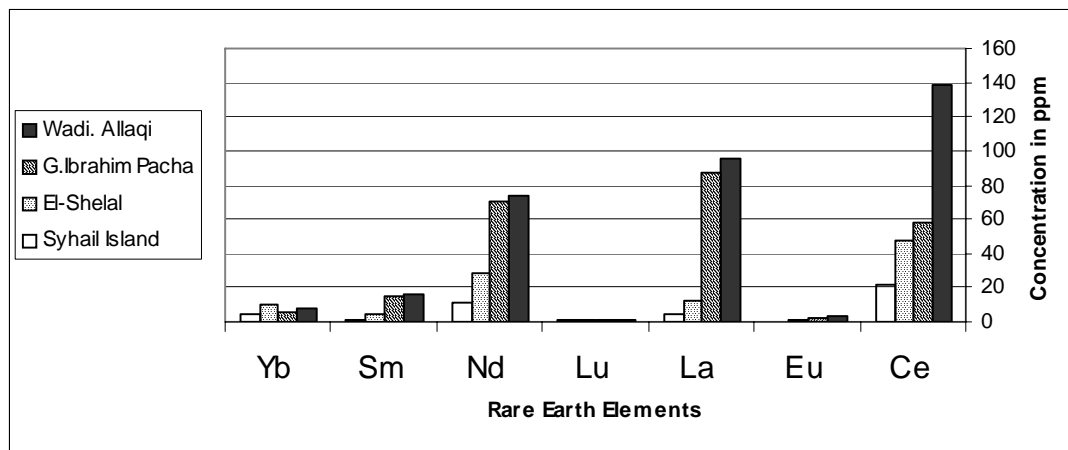


Figure 3. The concentration of the rare earth elements in the studied areas

CONCLUSION

The study of REE concentrations in rocks has long been appreciated as an important source of scientific information, which helps in predicting the source and evolutionary history of the rocks. The results obtained indicate the viability of using the INAA for the determination of the elements La, Ce, Nd, Sm, Eu, Yb and Lu. The results show that the concentration of the determined REEs in the old granite (Wadi El-Allaqi and Gebel Ibraheem pacha) are higher than that the same elements in the young granite (El Shalal and Seyhel Island). The high concentrations in the old granite related to the following factors: a) The presence of radioactive accessory minerals such as uranothorite; b) The granitic rocks contain accessory minerals such as Zircon, Sphene and also monazite and apatite which readily accumulate in limited uranium and thorium. The levels of the rare earth elements in granite samples of selected sites in south Egypt differed depending on their origin and geochemistry. The INAA technique can be effectively used on a routine basis for the analysis of a large number of samples in ore prospect and geochemical studies.

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