

Vol 8(1), 13-28 Copyright © Madda Walabu University ISSN: 2523-1901 (Online) URL: <u>http://jessd.mwu.edu.et/</u> Email: <u>JESSDeditorials@mwu.edu.et</u>



DOI: https://doi.org/10.20372/mwu.jessd.2025.1566

Full Length Research Paper

Abstract

Assessment of Elemental Compositions in the Soils of Hakim Gara Quarry Using Instrumental Neutron Activation Analysis

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Article Info

Article History Received: 25 Nov 2024 Accepted: 26 Jan 2025

Keywords:

Elemental concentration; INAA; Irradiation; GHARR-1 Research reactor

The fast, accurate, and non-destructive analytical tool known as Instrumental Neutron Activation Analysis (INAA) was employed to qualitatively and quantitatively assess major, minor, and trace elements in twenty soil samples collected from the Hakim Gara quarrying area in Ethiopia. The samples were irradiated at the Ghana Nuclear Research Reactor (GhIRR-1). This technique identified a total of 24 elements, including seven rare earth elements (REE): Al, Fe, K, As, Br, Ce, Co, Cr, Cs, Eu, Ga, La, Mn, Na, Rb, Sb, Sc, Sm, Ta, Tb, Th, U, V, and *Yb. The study compared the measured values of elemental concentrations in two groups (I* and II), revealing that the elemental concentrations were generally higher in group I than in group II. The concentrations of the identified elements ranged from 0.83 mg/kg for Sb to 5.4% for Al. The concentrations of the long-lived radionuclides U, Th, and K were measured as 0.7 ± 0.2 , 8.5 ± 4.1 , and 3.0 ± 1.0 , respectively. The average elemental concentrations obtained in this study were compared with similar studies, the continental upper crust, and global averages. The highest concentration values were found for Ga, Mn, Na, and Tb, while the lowest value was measured for Yb. The concentrations of K and Th were found to be lower than those in the continental crust, whereas the concentration of U was slightly higher. Further research should be conducted in quarry sites throughout the country.

Abdelhalim, 2014).

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1. Introduction

Expansion of urbanization around cities and towns, if not controlled properly, could elevate the level of pollution in an unpredictable manner. Toxic and radiation emitted from different sources of radionuclides is one form pollution that threatens human beings. Radiation exists ubiquitously and found in all forms of matter (including in soils) formed from radionuclides of Earth's crust, cosmogenic or anthropogenic sources. Anthropogenic and the naturally occurring processes such as weathering of rock and volcanic eruptions plays a significant effect in enriching the ecosystems with metals (El-Taher &

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Radioisotopes found in the soil samples are categorized as short-lived isotopes (Mg, Al, Ti, V, Ba, Eu), intermediate lived isotopes (Na, K, Mn, Dy) and long-lived isotopes (Sc, Cr, Fe, Co, Ce, Rb, La, Hf, Sm, As) (Essien et al.,2016). Since the constituents of the soil formed depends on the base foundation rock, man-made activities such as mining may accelerate the movement of primordial radionuclides into soil surface (Radtown, 2006). The types, amounts, and distributions of these nuclides in the environment, needs qualitative and quantitative assessment for their contribution of radiation exposure.

Besides exploring deposited minerals, quarrying activity is one of the ancient activities on limestone and granite for construction and related purposes. Even though the benefit of quarrying activity for development is undeniable, its impact on the environment is one aspect that still needs to be assessed. Mining has been associated with exposing radionuclides and heavy toxic elements contained in earth's crust to the soil surface (Atambo, 2011). It is also reported by United Nation Scientific Committee on Effects of Atomic Radiation (UNSCEAR) that mining is one of the activities that enhances human's exposure to the natural background radiation (UNSCEAR, 2000). At quarries, the rock may uncover radionuclides and heavy toxic elements that could expose quarry workers to health hazards due to terrestrial ionizing radiation sources (Ruth et al.,2020).

Environmental monitoring involves all forms of matter. Soil is a complex mixture of various components and carries many elemental compositions. It is one entity in the environment which is formed through gradual and different processes (like weathering of parent rock, translocation, erosion, etc.) in the terrestrial. The compositions of soil can be analyzed using different methods, among which Neutron Activation Analysis (NAA) is used to assess the different types and levels of the constituents. NAA is one of the most commonly used specific and accurate analytical techniques for analyzing elemental compositions of a sample qualitatively and quantitatively (Toma et al.,2008; Essien et al.,2016; Das et al.,2018). It is a method to determine mass fractions of the chemical elements by inducing radioactivity through neutron irradiation and detecting the emitted radiations (IAEA, 2023). NAA has vast applications in Chemistry, Geology, Geophysics, Archaeology, Environmental Impact Assessment, Forensic Science as well as Medicine and it is mostly preferred to be used for samples with high contents of radionuclides to several methods because of its non-destructive nature, very simple and fast method of analysis (Omeje et al.,2019). From the existing different analytical methods: it has a potential that enables to obtain large number of elements at a time (Ene et al.,2011), since it requires minimum preparation and because it is effective and non-destructive. Instrumental NAA (INAA) analytical method was employed for this study to determine the elemental makeup of soils.

NAA works by the principle of a neutron-gamma (n, γ) reaction which is considered the interaction of neutron with a nucleus. The neutrons are categorized according to their energies as thermal (< 0.5 eV), epithermal (0.5 eV - 0.5 MeV) and fast (> 0.5 MeV) (Kpeglo et al., 2012; Heilbronn, 2015). When a neutron bombards the target nucleus in a non-elastic collision, an excited state compound nucleus will be formed which will instantaneously de-excite into a more stable configuration through emission of one or more characteristic Prompt gamma rays (On-beam) in which measurements should be taken while irradiation takes place (Glascock, 1996). In many cases, this new configuration yields a radioactive nucleus which also decays by emission of one or more characteristic Delayed gamma rays (Off-beam) in which measurements taking place after irradiation (Glascock, 1996; Ali, 1999; CERAD, 2021). It is described in the International Atomic Energy Agency technical document that the major, minor, and trace elements contained in the samples can be identified both qualitatively and quantitatively from the gamma radiation emitted by the sample's induced radioactivity (IAEA-TECDOC-564). As it is also mentioned in (CERAD, 2021), because of its high sensitivity, NAA is used to examine minor elements, particularly trace elements that are present in very low concentrations. This study followed the single comparator standard method with known concentration of elements simultaneously irradiated along with the sample under similar experimental

conditions (Kpeglo et al.,2012) to ascertain concentrations of elements from a sample, rather than the absolute method. The analysis was based on gamma radiation measurement emitted from the irradiated samples in the research reactor center. The rate at which gamma rays are emitted from the element of the sample is directly proportional to the concentration of that element (CERAD, 2021).

Similar studies were done in different countries by (Ene et al.,2011; El-Taher & Abdelhalim, 2014; Elias et al.,2017; Onoja & Ewa, 2020). In Ethiopia also, studies were conducted by number of researchers (Endalew, 2019; Haileyesus Walle et al.,2000; Gashaw & Aklilu, 2020; Birhane et al.,2020; Gudissa et al.,2021; Asmamaw et al.,2021) and others. All of the assessments or studies have focused on impacts of dust contamination from quarrying on nearby water bodies, vegetation and people working and living around. Previous studies, in and around the stone quarry sites of the study area, did not take into consideration the identification and determination of the elemental composition as well as the concentration of naturally occurring radionuclides using the NAA technique.

The aim of this study was to fill this gap and to carry out identification of elements and measuring elemental concentration in the soil samples of Hakim Gara quarrying areas (in Harari region of Ethiopia) using NAA technique and also to analyze the low-level energy radionuclides U, Th and K. The result would be an input to the safety issues regarding natural radiation exposure situations from the quarrying operations and provide empirically evidenced guidance for future quarry management.

2. Materials and methods

Description of the Study area

The quarrying activity site on mount Hakim Gara, at about 5 km from Harar town in Ethiopia, is located from 9.2840 N to 9.2860 N and from 42.1020 E to 42.1180 E. Previous study showed deposits of large limestone formed the geology of the area and people are extracting it as commercial-sized blocks and supplying to the town as well as input for marble industry (Haileyesus Walle et al.,2000). In the study area, stones are

extracted as cobblestones for road and pavement works, as input for building houses and for other construction purposes. Trucks are moving the products to the surrounding areas and create dust in the environment and along the graveled road on which people are living. In some of the abandoned quarrying sites people started settling and turning the area to a residential place.



Figure 1: The study area map

The materials used in this study included a grinder with metal balls, a sieve, a plastic sheet, plastic capsules, a hot glue gun, an electronic balance, a research reactor, and an HPGe gamma spectrometry system.

Sampling

Before collecting the samples, unwanted parts on the surface of the undisturbed soil like roots and organic parts were removed. A total of twenty soil sample locations were randomly selected and surface soil samples were taken from Hakim Gara quarry sites during the dry season of March in 2023. The soil samples were taken from the depths of 5, 10 and 15 cm, mixed, each sample was transferred into separate clean plastic bag, and the bags were labeled.

Since the sites are varied in longitude and altitude and mountainous, two groups were formed as shown in Table 1 based on their altitudinal locations. Group I sample locations lies between 42.1129 E and 42.1202 E with altitudes ranging from 2046 m to 2126 m and group II sample lies between 42.4227 E and 42.1302 E and the altitudes range from 1991 m to 2045 m. The mean altitude of Group I sample locations is higher than the mean altitude of group II sample locations. Due to the hilly topographic of group I it was challenging to dig that made differences in the sample sizes between the two groups (Regassa *et al.*,2023).

Table 1: Geographical L	ocations of Two	Groups at Hakim	Gara Quarry Sites
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Group	Sample code	Lat. (N)	Long, (E)	Alt. (m)
	TS1	9.2992	42.1129	2046
	TS2	9.2874	42.1202	2122
	TS3	9.2991	42.1131	2049
	TS5	9.293	42.1149	2070
Ι	TS9	9.2865	42.1201	2126
	TS16	9.2932	42.1153	2070
	TS17	9.2928	42.1149	2070
	TS18	9.2868	42.1196	2126
	TS 20	9.3501	42.1664	2121
Group mean	1			2088.9

Group SD				32.3
Group CV				0.015
	TS4	9.2953	42.123	2026
	TS6	9.2969	42.1302	1958
	TS7	9.2958	42.1231	2018
	TS8	9.2965	42.1228	2009
II	TS10	9.2962	42.1228	2014
	TS11	9.2969	42.1229	2009
	TS12	9.2948	42.1232	2016
	TS13	9.2958	42.1227	2022
	TS14	9.2964	42.1233	1991
	TS15	9.2901	42.128	2021
	TS19	9.2959	42.1227	2024
Group mean	1			2009.8
Group SD				18.8
Group CV				0.009

Sample preparation

The sample preparation involved drying, grinding and sieving of the samples. Though samples were collected during the dry season, the soil samples were further allowed air dried for about 5 days in the laboratory. Then the samples were ground using metal balls mill and sieved with 450 µm mesh wire to homogenize and remove unwanted residuals.

For sample source preparation and elemental analysis, each sieved sample were taken to the Research Reactor-1 (GHARR-1) NAA laboratory of Ghana Atomic Energy Commission (GAEC). The soil samples were separately weighed (about 150 mg each), wrapped in clean transparent polyethylene film, given codes and sealed with heat. After placing the wrapped samples in a polyethylene irradiation capsule of cylindrical-shaped (diameter 1.6 cm and height 5.5 cm), the capsules were heat-sealed and made ready for irradiation.

The samples were packaged for short, long, and medium-lived radioisotope irradiations. For the medium and long-lived radioisotopes, the gold comparator (Al-0.1%Au wire from Institute for Reference Materials and Measurements of the European Commission's Joint Research Centre) was used as both reactor thermal flux monitor and single comparator element in the case of k0method sandwiched between the wrapped samples. The sample and comparator were then packaged together into polyethylene vials (rabbit capsule) for irradiation (Baidoo *et al.*,2014). Equal masses of standard soil reference materials (ISE) were prepared in the same way as the soil samples and were used concurrently as comparator reference standard and control. A control capsule with no sample (only cotton inserted) was heatsealed and prepared for the background analysis (Kpeglo *et al.*,2012; Kpeglo *et al.*,2015).

Sample Irradiation and Counting

The working principle of NAA applied in this study is a neutron-gamma (n,γ) reaction which is considered the interaction of neutron with a nucleus capture cross-section probabilistic. The nuclide type determines the relationship between neutrons energy and the capture cross section. Neutrons are the activating particles that induce different kinds of nuclear reactions based on their energy (Osie, 2002). This study followed the single comparator standard method with known concentration of elements simultaneously irradiated along with the sample under similar experimental conditions (Kpeglo *et al.*,2012) to ascertain concentrations of elements from a sample, rather than the absolute method.

The standard and soil samples prepared were transferred to the irradiation site core through automatic pneumatic transfer system A (automated handling of sample called *rabbit console* working with 0.6 MPa pressure). The irradiations were carried out in the inner pneumatic irradiation sites A1 of the GHARR-1 center. The reactor used Low-Enriched Uranium (LEU) (13%) as a neutron source and light water as a moderator and coolant. The tank-in-pool Miniature Neutron Source reactor was routinely operated at half full power of 17 KW with a maximum thermal flux of 5×1011 n.cm⁻² s⁻¹ (Baidoo et al., 2013; Osei et al.,2021). The neutron beam from a reactor consists of thermal, epithermal and fast neutrons. The neutron beam has to be thermalized because most often the thermal neutrons are required for NAA due to their maximum absorption crosssections. A control system is connected via each channel for the purpose of neutron flux monitoring during samples irradiations (Hailu, 2018). In the NAA method in which irradiated stable elements with thermal neutrons in a nuclear reactor become radioactive due to the neutron capture of the core, element determination is based on measurement of characteristic gamma energies emitted from artificially produced radionuclides

(CERAD, 2021).

The schemes for irradiation and counting data for the elements of interest, as presented in Table 2, were based on the IAEA nuclear data and references (Baidoo et al., 2014; Onoja & Ewa, 2020). The samples were irradiated under three categories: for short-lived radionuclides (half-life less than 5 hours), for medium-lived radionuclide elements (half-life between 5 hours and 7 days) and for long-lived radionuclides (half-life more than 7 days). To analyze the constituent elements in each sample, the irradiation scheme was applied by optimizing irradiation time (t_i) , decay time (t_d) and counting time (t_c) based on the half-life $(t_{1/2})$. Hence t_i, t_d and t_c for long-lived, medium-lived and short-lived lived were (5 h, 7 d, 2 h), (2 h, 2 d, 15 min) and (1 min, 5-10 min, 600 s), respectively.

1abic 2.1	The first and the fermionic used to unaryze elemental composition of son by (n, j) reaction										
Targeted Element	Product nuclide by (n,γ)	Gamma En- ergy (keV)	Abund- ance (%)	Cross- section (barn)	Half-life	Irradiation by category	Irradia- tion time (t _i)	Delay time (t _d)	Counting time (t _c)		
²⁷ Al	²⁸ Al	1778.99	100	0.23	2.24 min	SL	1 min	5-10 min	600 s		
⁵⁸ Fe	⁵⁹ Fe	1099.25	0.28	1.31	44.5 d	LL	5h	7d	2h		
⁴¹ K	⁴² K	1524.6	0.06	1.45	12.36 h	ML	2h	2d	15min		
⁷⁵ As	⁷⁶ As	559.1	100	3.86	26.32 h	ML	2h	2d	15min		
⁸¹ Br	⁸² Br	776.52	49.31	2.58	35.3 h	ML	2h	2d	15min		
¹⁴⁰ Ce	¹⁴¹ Ce	145.4	88.5	0.575	32.5 d	LL	5h	7d	2h		
⁵⁹ Co	⁶⁰ Co	1173.2	100	37.13	5.27 y	LL	5h	7d	2h		
⁵⁰ Cr	⁵¹ Cr	320.1	4.35	15.2	27.7 d	LL	5h	7d	2h		
¹³³ Cs	¹³⁴ Cs	795.8	100	30.3	2.06 y	LL	5h	7d	2h		
¹⁵¹ Eu	¹⁵² Eu	1408.5	47.8	5900.0	13.3 y	LL	5h	7d	2h		
⁷¹ Ga	⁷² Ga	603.02	40	4.61	3.3 d	ML	2h	2d	15min		
¹³⁹ La	¹⁴⁰ La	1596.2	99.91	9.34	40.3 h	LL	5h	7d	2h		
⁵⁵ Mn	⁵⁶ Mn	846.8	100	13.2	2.58 h	SL	1min	5-10 min	600 s		
²³ Na	²⁴ Na	1368.6/2754	100	0.513	15 h	ML	2h	2d	15min		
⁸⁵ Rb	⁸⁶ Rb	1076.6	72.2	0.494	18.7 d	LL	5h	7d	2h		
¹²³ Sb	¹²⁴ Sb	1691	47.34	4.08	60 d	LL	5h	7d	2h		
⁴⁵ Sc	⁴⁶ Sc	889.3	100	26.3	83.8 d	LL	5h	7d	2h		
¹⁵² Sm	Sm	104.35	26.7	220.0	22.3 min	SL	1 min	5-10 min	600 s		
¹⁸¹ Ta	¹⁸² Ta	1221.4	100	20.4	114.4 d	LL	5h	7d	2h		
¹⁵⁹ Tb	¹⁶¹ Tb	74.58	100	23.8	6.9 d	ML	2h	2d	15min		
²³² Th	²³³ Pa	312.1	100	7.26	27 d	LL	5h	7d	2h		
²³⁸ U	²³⁹ Np	277.6	99.27	2.75	2.36 d	ML	2h	2d	15min		
⁵¹ V	⁵² V	1434.1	99.75	4.79	3.76 min	SL	1 min	5-10 min	600 s		
¹⁷⁴ Yb	¹⁷⁵ Yb	396.3	31.8	128.0	4.19 d	LL	5h	7d	2h		

Table 2. Nuclear data of elements used to analyze elemental composition of soil by (n,γ) reaction

SL = short-lived, ML = medium lived, and LL = long-lived

Measurements and Data collection

Measuring of the gamma energy produced from

the induced radionuclides formed were carried out after the nuclear reaction using a gamma ray spectrometry technique of high-resolution HPGe detector n-type high purity germanium (HPGe) detector of model GEM-C5970 manufactured by Canberra, Coaxial type, and coupled with Multichannel analyzer (MCA) connected to a PC. The detector is mounted in a cylindrical leaded shield and kept cooled with the supply of liquid nitrogen. The detector, with resolution of 2.0 KeV (FWHM) for 60Co gamma-ray energies of 1332.4 KeV, operated with relative efficiency of 40%. The induced radionuclides produced from INAA were identified by their energies using the nuclear data taken from (IAEA, 1990) as shown in Table 1. The γ -ray emitting radionuclides were qualitatively identified by the energies emitted and analyzed quantitatively the concentration of elements from the obtained γ -ray spectrum. The areas under photo peaks of each radionuclide were converted to counts and used for quantitative analysis by the single comparator ko-method for the same geometry, equal weights of both sample and standard, as well as through appropriate choice of cooling time, the detector's dead time and counting times. Qualitative analysis was done using γ -ray acquisition software, (Genie 2000 Gamma Acquisition and Analysis software) and quantitative analysis using ko-IAEA the spectrum analysis software

Calculating Activity and Analyte concentration

Many elements can be quantitatively measured at a time when counting of a sample is performed with germanium detector associated with the INAA. The amount of radioactive nuclide formed when a sample is irradiated can be calculated as (Adamson, 1979),

$$R = \varepsilon \varphi \sigma N (1 - e^{-\lambda t_{irr}}) e^{-\lambda t_{cool}} \cdot (1 - e^{-\lambda t_c}) \lambda^{-1} \quad (1)$$

where: *R* is the amount of radionuclide formed, ε is efficiency of the measuring apparatus. φ is the bombarding flux, σ is the reaction cross-section, *N* is the number of target atoms of an element in the sample. *t_{irr}* is the irradiation time (up to saturation activity), λ is the decay constant, and *t_{cool}* is the time from end of bombardment to start of counting and *t_c* is time of counting (Adamson,

1979).

Concentration of elements in a sample can be determined using Equation (2) developed by Decorte *et al.* for k_0 factor (Joseph *et al.*,2017).

$$k_o = \frac{M_C \theta_a \sigma_a \gamma_a}{M_a \theta_C \sigma_C \gamma_C} \quad (2)$$

Where the subscripts, *a* is analyte and *c* is comparator. *M* is the atomic mass in (g/mol), θ is isotopic abundance σ is as defined above in Equation (1), and γ is absolute gamma-ray abundance. Concentration of elements in the soil samples can also be determined by comparing with known masses and concentrations of elements in a standard reference material using Equation (3) as defined in (Choudhury et al., 2007; Tsegaye Birhanu *et al.*, 2015).

$$C_{samp} = C_{std} \frac{A_{samp}}{A_{std}} \frac{(e^{-\lambda t_d})_{std}}{(e^{-\lambda t_d})_{samp}}$$
(3)

 C_{samp} and C_{std} are concentrations of unknown elements in the soil samples and in the standard; A_{samp} and A_{std} are the activity rates of unknown analytes in the sample and the standard. t_d is the decay time of the unknown element in the sample and in the standard, $(e^{-\lambda t_d})_{samp}$ and $(e^{-\lambda t_d})_{std}$ are the decaying factors for the analyte and the standard, respectively.

To compare the results due to the techniques the specific activities of ²³⁸U, ²³²Th, and ⁴⁰K obtained by Gamma spectrometry conversions into elemental concentrations in terms of masses for U, Th, and K, respectively were done using Equation (4) (Kpeglo *et al.*, 2012).

$$C_e = \frac{RM_a A_{sp} T_{1/2}}{N_a P_a \ln 2}$$
(4)

C_e is the elemental concentration in sample, M_a is the atomic mass (kg/mol), T_{1/2} is the half-life (sec), P_a is the fractional atomic abundance in nature (%), N_a is Avogadro's constant (6.023×10^{23} g/mol), A_{sp} is the measured activity concentration (Bq/kg) of the radionuclide considered (²³⁸U, ²³²Th, or ⁴⁰K), and *R* is a constant with a value of 10⁶ for U and Th (concentration in mg/g) or 10² for K (concentration in % of mass fraction).

3. Results and Discussions

The quantitative and qualitative analysis results obtained by the NAA technique and analysis of the spectrum performed on two groups of soil samples collected from the different locations of Hakim Gara stone quarrying area is displayed in Table **2** and Table **3**.

Table 2: Concentrations of element measured in soil samp	ples of group I collected from the study area
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Targeted			Concen	trations of	elements	measure	d in Samj	oles		Group	Group	Group
Element	TS1	TS2	TS3	TS5	TS9	TS16	TS17	TS18	TS 20	mean	SD	CV
²⁷ Al	8.34	4.73	6.81	9.12	8.56	8.03	9.17	9.5	9.13	8.2	1.4	0.18
⁵⁸ Fe	6.65	8.37	4.19	7.45	7.17	7.77	7.4	8.0	8.49	7.3	1.2	0.17
⁴¹ K	1.17	0.82	1.14	0.71	0.69	0.81	0.67	0.66	0.76	0.8	0.2	0.22
⁷⁵ As	11.3	15.7	6.3	14.2	13.6	13.4	13.8	15.5	16.2	13.3	2.9	0.21
⁸¹ Br	39.9	7.2	31.8	45.1	8.8	15.7	45.6	26.9	7.9	25.4	15.1	0.59
¹⁴⁰ Ce	118	110.1	54	106.5	87.2	141.4	107.6	93.2	104	102.4	22.4	0.22
⁵⁹ Co	31.7	40.6	19.9	38.2	32.9	49.5	40.5	35.1	32.9	35.7	7.6	0.21
⁵⁰ Cr	133	233	110	217.1	230.4	228.2	216.2	277.2	269	212.7	52.9	0.25
¹³³ Cs	5.7	6.6	3.8	6.3	5.4	6.1	5.2	6	5.4	5.6	0.8	0.14
¹⁵¹ Eu	3.4	3	2.2	2.8	2.8	2.6	2.9	2.9	3	2.8	0.3	0.11
⁷¹ Ga	23.6	29.6	16	28.3	24.9	28.3	27.2	28.9	28.1	26.1	4.0	0.15
¹³⁹ La	44.4	59.5	28.9	54.2	49.2	56.2	52.6	52.7	61.3	51.0	9.2	0.18
⁵⁵ Mn	1159	688.7	904	1273	1021	1647	1422	993.7	1310	1157.6	274.0	0.24
²³ Na	982	671.1	841	980.5	1188	794.1	1108	632.9	574	863.5	203.3	0.24
⁸⁵ Rb	95.5	97.3	69.7	77.2	71.1	83.9	77.3	85.7	80	82.0	9.1	0.11
¹²³ Sb	1.1	1.2	0.6	1.1	1	1	1.2	1.5	1.2	1.1	0.2	0.21
⁴⁵ Sc	21.2	26	13.3	23.5	23.7	24.4	23.2	25.5	27.5	23.1	3.9	0.17
¹⁵² Sm	7.3	9.4	4.6	8.5	7.4	8.8	8.4	8.6	10	8.1	1.5	0.18
¹⁸¹ Ta	BDL	2.5	1.4	1.9	1.8	2.4	2.2	2.5	3.1	2.2	0.5	0.22
¹⁵⁹ Tb	1.5	1.2	1	1.2	1.5	1.2	1.5	1.4	1.2	1.3	0.2	0.13
²³² Th	13.1	13.7	7.3	13.7	12.5	13.4	11.8	8.3	14.6	12.0	2.4	0.20
²³⁸ U	3.6	2.8	BDL	3.9	BDL	2.5	BDL	5.1	4	3.7	0.9	0.23
⁵¹ V	184	88.6	161	190.4	179.7	161.8	199.8	176.4	190	170.2	31.3	0.18
¹⁷⁴ Yb	4.2	4.3	2.8	4.3	4.5	5.1	BDL	5.1	5.4	4.5	0.8	0.17

Table 3: Concentrations of element measured in soil samples of group II collected from the study area

Targeted Floment			(Concentra	tions of e	lements n	neasured i	in Sample	s			Crown moon	Crown SD	Group CV
Targeteu Element	TS4	TS6	TS7	TS8	TS10	TS11	TS12	TS13	TS14	TS15	TS19	Group mean	Group SD	
²⁷ Al	2.38	3.09	6.32	5.59	0.44	4.84	4.87	0.25	0.46	3.82	2.8	3.2	2.0	0.65
⁵⁸ Fe	1.69	2.28	5.49	3.5	5.29	3.67	3.85	2.33	3.23	2.88	2.98	3.4	1.1	0.33
⁴¹ K	0.56	0.42	0.92	0.59	0.75	0.71	0.76	0.57	0.71	0.74	0.59	0.7	0.1	0.19
⁷⁵ As	2.9	4	9.6	5.9	8.1	6.3	6.9	3.9	5.2	5.4	5.1	5.8	1.8	0.32
⁸¹ Br	20.6	41.4	69.2	37.9	38.6	33.1	50.7	34.6	33.9	25.2	46.5	39.2	12.5	0.32
¹⁴⁰ Ce	19.8	35.6	86.5	58.2	73.3	60.5	48.3	27.7	42.7	42.7	34.3	48.1	19.0	0.40
⁵⁹ Co	8.1	11.1	26.1	18.5	24.5	18.9	20	11.1	15.5	14.3	14.5	16.6	5.4	0.32
⁵⁰ Cr	40.3	63.3	135	89.9	134.9	102.1	104.4	62.2	90.6	77.2	78.5	88.9	28.0	0.31
¹³³ Cs	1.2	1.9	4.3	2.9	4.3	3	3.3	1.9	1.9	2.7	2.5	2.7	0.9	0.35
¹⁵¹ Eu	1.2	1.5	2.7	2.2	2.1	1.7	1.8	2.2	1.8	1.6	1.6	1.9	0.4	0.21
⁷¹ Ga	7.1	8.6	19.1	11.8	17.3	13.6	14.3	9.6	12.2	12.3	11.8	12.5	3.4	0.27
¹³⁹ La	11.2	14.1	38.6	26.6	32.3	25.9	26.9	15.7	22.6	19.9	20.6	23.1	7.7	0.33
⁵⁵ Mn	318.2	434.9	1066	686.6	69.7	868.4	743.7	42	83.7	587.3	448.8	486.3	325.3	0.67
²³ Na	498.4	639.9	1167	894.9	1303	1308	991.4	685.9	1309	879.6	1019	972.4	271.0	0.28
⁸⁵ Rb	30.7	30.2	81.1	45.1	52.8	62.7	63.4	34.2	48.9	48.1	46	49.4	14.7	0.30
¹²³ Sb	0.2	BDL	0.7	0.5	0.7	BDL	0.4	0.5	BDL	0.4	BDL	0.5	0.2	0.34
⁴⁵ Sc	5.5	7	17.4	11.1	15.8	11.9	12.3	7.6	10.5	9.7	9.6	10.8	3.4	0.32
¹⁵² Sm	1.6	2.2	6.3	4.2	5.1	4.2	4.8	2.5	3.5	3	3.1	3.7	1.3	0.36
¹⁸¹ Ta	0.5	0.7	1	0.9	1.2	1.1	1.1	BDL	1	0.7	0.8	0.9	0.2	0.23
¹⁵⁹ Tb	0.4	0.3	1.1	0.8	0.8	0.7	0.6	BDL	0.6	0.5	0.9	0.7	0.2	0.34
²³² Th	1.5	2.3	10.4	6.6	8.9	7.2	6.4	7.1	2.8	4.2	4.5	5.6	2.7	0.48
²³⁸ U	2.1	BDL	3.8	BDL	2.4	2.1	BDL	1.5	BDL	2.5	BDL	2.4	0.7	0.29
⁵¹ V	46.6	65	145.3	BDL	10.4	95.5	102.6	4.9	9.9	79.3	64	62.4	43.5	0.70
174 Vb	0.8	11	3.6	0.2	33	2	23	1.6	19	11	1.8	1.8	1.0	0.54

In both groups of soil samples, all the twentyfour trace, minor and major elements Al, Fe, K, As, Br, Ce, Co, Cr, Cs, Eu, Ga, La, Mn, Na, Rb, Sb, Sc, Sm, Ta, Tb, Th, U, V and Yb were identified. Of these identified elements, the major, minor and trace elements (by their percentage constituent in the sample: > 1% for major, 0.1 to 1% for minor and < 0.1% for trace (Pendias, 2010)) based on their concentrations, are (K, Al, Fe, Mn, Na), (Ce, Cr, Rb,

V) and (As, Br, Co, Cs, Eu, Ga, La, Sb, Sc, Sm, Ta, Tb, Th, U, Yb.), respectively.

In group I samples as presented in Table 3a, the values of CVs were found in ranges of 10 - 20% for 13 samples, 21 - 30% for 10 samples and > 30% for 1 sample. These tell that the spread of majority of the data of the elements are around their similar element's mean value of group I and only poor spread of one element was observed which means it is far from similar element's mean in this group.

In Table 3 of group II elements, the CV value of each element is in the range of 10 - 20% for one sample, 21 - 30% for 6 samples and > 30% for the rest of the (17) samples. In group II, the spread of 7 elements from their group mean are close and the rest 16 are poorly spread which means they are far from the mean values of elements in the group. The values of six elements are spread moderately from the mean of the group, while one element showed wide spread from the mean.

Of the total twenty-four elements identified in group I, 22 elements (92%) exhibited maximum elemental concentrations and 2 elements (8%) minimum readings were observed from group I and vice versa. Out of the 24 identified elements, the maximum elemental concentration readings of eight elements (Fe, As, La, Sc, Sm, Ta, Th, and Yb) were from soil sample TS20 of group I.

Though similar, the result of elemental concentrations measured for the long-lived radionuclides U, Th and K, were analyzed separately and compared between group I and group II as shown in Table 3 and Table 4. The comparison results of the two groups shows that average concentrations of U, Th and K measured are 3.7 ppm, 12.0 ppm and 0.8% in group I and 2.4 ppm, 5.6 ppm and 0.7%, in group II, respectively. This revealed the U, Th and K concentration are found higher in group I than the values in group II. In both groups average concentrations of ⁴⁰K are much lower whereas the ²³⁸U and ²³²Th values are observed higher than the continental crust average value (Chaffee & Berry, 2006; Rudnick & Gao, 2013),.

The results of U, Th and K elemental concentrations obtained using INAA method of this study were compared with those of the results obtained for radioactivity concentration using Gamma Spectrometry (GS) method as reported in our previous study (Regassa *et al.*,2023) as shown in Table 4.

Table 4: Maximum and minimum measured values of U, Th and K by INAA and Gamma spectrometry in samples.

INAA							Gamma spectrometry (Regassa et al., 2023)				
Ele- ment	Max	Group	Min	Group	Average (I, II)	Max	Group	Min	Group	Ave. (I, II)	
U	TS18	Ι	TS13	II	3.7, 2.4	TS18	Ι	TS15	II	68.3, 38.4	
Th	TS20	Ι	TS4	II	12, 5.6	TS16	Ι	TS4	II	94.9, 46.5	
Κ	TS1	Ι	TS6	II	0.8, 0.7	TS1	Ι	TS6	II	242.7, 201.4	

The majority of maximum and minimum values measured for the two techniques are from the same group and sample locations. As indicated in the table all the elemental concentrations measured by INAA and activity concentrations measured by GS for the three long lived radionuclides U, Th and K were found to be higher in group I and lower in group II as shown in Table 4. As the mean values of U, Th and K in group I are higher than the mean values in group II for the GS technique, the mean values of the same elements in group I are higher than mean values in group II for INAA technique. Comparison of the results obtained by the two methods has shown the two methods agree.

The values of mean elemental concentrations of each element in the samples of the two

groups (group I and group II, based on their altitudes) were compared and are presented

in Table **5** and Figure 2: Mean values of each elemental concentration in the two groups (I and II) soil samples

Flomont	Group I	Group II		Flomont	Group I	Group II	
Liement	Average	Average	I/II	Element	Average	Average	I/II
Al (%)	8.2	3.2	2.6	Mn (%)	1157.6(0.12)	486.3(0.05)	2.4
Fe (%)	7.3	3.4	2.1	Na (%)	863.5(0.09)	972.4(0.1)	0.9
K (%)	0.8	0.7	1.1	Rb	82	49.4	1.7
As	13.3	5.8	2.3	Sb	1.1	0.5	2.2
Br	25.4	39.2	0.6	Sc	23.1	10.8	2.1
Ce	102.4	48.1	2.1	Sm	8.1	3.7	2.2
Co	35.7	16.6	2.2	Ta	2.2	0.9	2.4
Cr	212.7	88.9	2.4	Tb	1.3	0.7	1.9
Cs	5.6	2.7	2.1	Th	12	5.6	2.1
Eu	2.8	1.9	1.5	U	3.7	2.4	1.5
Ga	26.1	12.5	2.1	V	170.2	62.4	2.7
La	51	23.1	2.2	Yb	4.5	1.8	2.5

Table 5: Comparing mean elemental concentrations of the two groups on the basis of ratio of their means (I/II)

Comparing mean concentrations of elements in the two groups, except for six elements (Na, K, Eu, Rb, Tb, and U), the mean concentrations of elements in group I were more than two-fold higher from similar elements in group II. Mean concentration of elements (K, Na) in group I were about the same as in group II. Only the mean concentration of element (Br) in group I was found to be lower than those in group II. Except for K, Na and Br, the values of average concentrations of all elements of group I are twice and higher than the values in group II.



Figure 2: Mean values of each elemental concentration in the two groups (I and II) soil samples

The results of average values of elemental concentrations of U, Th and K obtained from

twenty samples of this study were compared with other countries studies as presented in T

Countries	Eler	nental concent	ration	Reference		
Countries	K (%)	Th (ppm)	U (ppm)			
USA (California)	2.2	17	4.2	(Chaffee & Berry, 2006)		
Egypt	4.8	9.7	2.7	(El-taher & Abdelhalim, 2015)		
Egypt (Assuit)	-	9.6	2.7	(El-taher & Abdelhalim, 2015)		
Ghana	1.25	7.8	3.6	(Kpeglo <i>et al.</i> ,2012)		
Continental Crust	2.8	10.5	2.7	(Rudnick & Gao, 2013; Chaffee & Berry, 2006)		
This study	0.74±0.2	8.52±4.1	3.03±1.0			

Table 6: Comparison of concentrations of K, Th and U of this study with other studies

As far as the naturally occurring long-lived radionuclides (K, Th, and U) concerned and as shown in Table 6, the amount of K concentration obtained in this study is lower from countries studies (one third the results of the study in USA, one sixth of the result of a study in Egypt, one half of result of a study in Ghana) and one-fourth of the amount of K concentration for the continental crust. As far as Th is concerned, the result we obtained is similar to the values of two African countries, namely Egypt and Ghana, and it is also consistent with continental crust. But the value of Th in this study is half of the amount of concentration from a study in USA. The amount of U concentration obtained in this study was almost the same as the results obtained in the studies from Egypt, Ghana and consistent with the continental crust, but, a bit lower than the result obtained from the study in USA. The results of average elemental concentrations obtained in this study were related with some similar studies of other countries as presented in Table 7: Mean elemental concentrations of this study compared with other countries similar studies.

From the results of this study, it was observed that the concentrations of elements measured using Neutron Activation Analysis (NAA) were higher for iron (Fe), arsenic (As), cerium (Ce), cobalt (Co), chromium (Cr), cesium (Cs), europium (Eu), gallium (Ga), lanthanum (La), manganese (Mn), antimony (Sb), scandium (Sc), samarium (Sm), terbium (Tb), uranium (U), vanadium (V), and ytterbium (Yb) compared to the values of the continental crust (Chaffee & Berry, 2006). In comparison with a study conducted in Egypt (El-Taher & Abdelhalim, 2015), the concentrations of cerium, cobalt, chromium, europium, lanthanum, manganese, scandium, samarium, and uranium were found to be higher in this study.

Additionally, a study in Ghana (D. Kpeglo et al., 2012) on Portland cement with 75% limestone reported higher concentrations of aluminum (Al), iron (Fe), potassium (K), arsenic (As), chromium (Cr), lanthanum (La), manganese (Mn), sodium (Na), thorium (Th), and vanadium (V) than those observed in this study. The concentrations of aluminum, cerium, manganese, and sodium in this study were also higher than those reported in a similar study conducted in Ethiopia (Hibstie et al., 2013).

When compared to research from the southeastern California region on soil (Chaffee & Berry, 2006), this study exhibited higher concentrations of iron, arsenic, cobalt, chromium, cesium, manganese, scandium, tantalum (Ta), vanadium, and ytterbium. Except for five elements—bromine (Br), cobalt (Co), chromium (Cr), europium (Eu), manganese (Mn), and terbium (Tb)—the results obtained in this study were generally consistent with global averages.

According to the 2003 Guideline of Ambient Environment Standards for soil in Ethiopia, the acceptable limits for arsenic (20 ppm) and chromium (20/500 ppm) (EEPA, 2003) indicate potential risks to human health. The concentrations of arsenic and chromium found in this study were higher than both the established safety guidelines and the global averages reported by A. K. Pendias (Pendias, 2010). These findings warrant careful consideration before utilizing the area for other purposes, due to the toxic potential of these heavy elements.

Element	This	Ethiopia	USA	Egynt	Egynt	Ghana	Continental	World
Element	Study	Etmopia	eon	Espe	Espr	Onunu	crust	Average
Al %	5.41	0.014	7.45	1.1		4.9	8.04	1 - 30
Fe %	5.13		3.82	0.8		0.4	3.5	3.5
K %	0.74		2.2	0.6	4.8	1.25	2.8	0.6
As	9.17		7.5		-	2.9	1.5	6.83
Br	33.03		-		-			10
Ce	72.60	118.7	100	17	3.38		64	56.7
Co	25.20		10	4	12	30.5	10	11.3
Cr	144.67		35		94	5.8	35	59.5
Cs	4.02		3.9		-		3.7	5.06
Eu	2.30		3		0.04		0.88	1.4
Ga	18.63		19		-		17	15.2
La	35.67		53	7	1.5	824.5	30	27
Mn%	788.40	6.5	680	128	0.8%	368.5	600	488
Na%	923.37	2.7	1.9%	1984	3.4%	0.03%	2.89%	
Rb	64.05		89		-		112	68
Sb	0.83		2.9	0.2	-		0.2	0.67
Sc	16.34		12	2	0.73	31	11	11.7
Sm	5.68		6.7		0.26		4.5	4.6
Та	1.49		1.4		-		2.2	1.39
Tb	0.97		-				0.64	0.63
Th	8.52		17		9.6	7.8	11	9.2
U	3.03		4.2		2.7	3.6	2.8	3.0
V	113.39		94	26	-	66	60	129
Yb	2.92	4.4	2.9		-		2.2	2.6
Reference	Hibstie e	et al.(2013)	Chaffee and Berry (2006)	Onoja and Ewa (2020)	El-taher and Abdelhalim, (2015)	Kpeglo <i>et al.(</i> 2012)	Chaffee and Berry (2006)	Pendias (2010)

Table 7: Mean elemental concentrations of this study compared with other countries similar studies

4. Conclusion and Implications

This study employed Instrumental Neutron Activation Analysis (INAA), one of the most powerful analytical techniques, to analyze soil samples from the Hakim Gara area. A total of twenty-four elements were identified, including major, minor, and trace elements, with seven classified as rare earth elements (REEs). The identified elements include aluminum (Al), iron (Fe), potassium (K), arsenic (As), bromine (Br), cerium (Ce), cobalt (Co), chromium (Cr), cesium (Cs), europium (Eu), gallium (Ga), lanthanum (La), manganese (Mn), sodium (Na), rubidium (Rb), antimony (Sb), scandium (Sc), samarium (Sm), tantalum (Ta), terbium (Tb), thorium (Th), uranium (U), vanadium (V), and ytterbium (Yb). Among these, the major elements such as Al, Fe, and K were present at percentage levels across all soil samples collected from the quarry area. The concentrations of the identified elements varied widely, ranging from 0.83 mg/kg for antimony to 5.4% for aluminum. The presence of long-lived radionuclides, such as uranium, thorium, and potassium, is significant for assessing their contributions to background radiation levels in the region.

The evaluation of elemental concentration distribution between two groups at different elevations in the Hakim Gara quarry revealed that, with the exception of bromine, soils at higher elevations generally exhibited greater concentrations of elements compared to those at lower altitudes. Given the limited information available on elemental analysis using the NAA method in stone quarrying areas, this study addresses a critical gap by providing valuable data regarding the elemental composition and concentrations, particularly of naturally occurring long-lived radionuclides.

The application of INAA for soil analysis in this study marks a first for the region and establishes a baseline for future investigations of soil and rock composition. The results can serve as a reference for ongoing environmental research, especially in geochemistry and related fields. Ethiopia's fastgrowing economy can benefit significantly from having a state-of-the-art research reactor capable of conducting various studies across multiple sectors, including mining, agriculture, and health. The INAA technique allows for rapid, accurate analysis of multiple elements from a single sample, thereby enhancing research capabilities and supporting evidence-based decision-making in these vital industries.

Acknowledgment

The author would like to acknowledge Ethiopian Technology Authority (ETA), International Atomic Energy Agency (IAEA) for sponsoring as the 18 months fellowship training, College of Natural and Computational Sciences of Haramaya University (HU) and School of Nuclear and Allied Sciences (SNAS) - University of Ghana. This experimental work was carried out at the Ghana Nuclear Research Reactor Center of Ghana Atomic Energy Commission (GAEC), and the author is thankful to all staff members of the ETA and Ghana Nuclear Research Reactor Center.

Author contribution statement

Tadele Negash Regassa: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed materials, analysis tools or data; Wrote the paper.

Gelana Amente Raba: Analyzed and interpreted the data; Contributed materials, analysis tools or data; Wrote the paper.

David Okoh Kpeglo: Performed the experiments; Analyzed and interpreted the data; Contributed materials, analysis tools or data; Wrote the paper.

Funding statement

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Conflicts of interest

The authors declare no conflicts of interest.

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