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Research Article

Elemental Analysis of Basalt by Instrumental Neutron Activation Analysis and Inductively Coupled Plasma Mass Spectrometer

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Abstract

Background: Because of its high sensitivity, activation analysis has become an important tool in a wide variety of science and engineering fields such as industry, mineral exploration, medicine, environmental monitoring and forensic applications. **Methodology:** Therefore, Instrumental Neutron Activation Analyses (INAA) Au+34 packages from ACT Lab Canada have been used to achieve accurate knowledge about the elemental analysis of basalt rock collected from Hail Northeast of Saudi Arabia. The samples were prepared for irradiation by thermal neutrons using thermal neutron flux of $7 \times 10^{12} \text{ n cm}^{-2} \text{ sec}^{-1}$. Twenty five elements were determined and identified namely: As, Co, Cr, Sb, Sc, Zn, Ba, Br, Sr, Zr, Cs, Hf, Mo, Rb, Th, U and eight rare earth elements namely: La, Ce, Nd, Sm, Eu, Yb, Tb and Lu. In addition, 15 elements were determined and identified by inductively coupled plasma mass spectrometer namely: Be, Cu, Dy, Er, Ga, Ge, Ge, Ho, Nb, Ni, Pb, Sn, Tm, V and Y. **Results:** The data presented here are our contribution to understanding the elemental composition of basalt rock. Because there are no existing databases for the elemental analysis of basalt, this results are a start to establishing a database for the basalt rock from Hail, Saudi Arabia. **Conclusion:** The acquired data was serve as a reference for the follow-up studies to assess the agronomic effectiveness of basalt rocks.

Key words: Elemental analysis, basalt, INAA, ICP-MS, ACT lab

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Data Availability: All relevant data are within the paper and its supporting information files.

INTRODUCTION

Nuclear analytical methods have been successfully applied to the determination of a great variety of elements in environmental, biological and geological samples. Actually different techniques could be used for estimating the trace, minor and major elements of these environmental samples, which are considered as complex samples. Neutron activation analysis techniques have been improved and have become an excellent tool for such purposes¹⁻⁵. Because nuclear reactions and decay processes are virtually unaffected by chemical and physical structure of the material during and after irradiation, the composition of the matrix has little influence on the induced activity put this technique in the forefront⁶.

The instrumental neutron activation analysis does not require sample dissolution with the consequent advantages that the analysis time and the risk of contamination from the reagents are greatly reduced. However, interference problems are often encountered, due to the overlapping of γ -ray lines and to the Compton continuum in the γ -ray spectrum. An approach to reduce this problem can be made by choice of a more convenient analytical peak, decay time and counting scheme. The major advantage of NAA are (a) The relative freedom from matrix effects and interferences, (b) High accuracy and (c) Very low zero blank contributions^{7,8}.

The application of INAA in geology permits the correlation of the elemental composition of productive and non-productive layers with either the presence or absence of radioactive or good for nuclear analysis isotopes⁹. The concentration of elements in basalt is poorly known. Therefore, the following are some of studies dealing with elemental analysis of geological rock samples in general.

Since basalt was widely used in industry and in scientific investigations, therefore, it is important to measure the concentrations of major, minor and trace elements in basalt rocks in addition to rare earth elements and the natural radionuclides uranium and thorium. These will be available for subsequent evaluations of the possible future environmental contamination due to human activity.

Basalts are the most abundant extrusive igneous rocks, which have erupted on earth throughout its history. Basalt as a general term is a fine-grained and mafic volcanic rock containing (44-54)% SiO₂. It consists essentially of calcic plagioclase and abundant mafic minerals mainly Ca-rich clinopyroxene, but may contain quartz, orthopyroxene, olivine, feldspathoid, small proportions of alkali feldspar (<10% total feldspar), Fe-Ti oxides and apatite¹⁰. Basalts are often associated with lava flows and narrow dykes and sills.

Most of basalts occur as lava flows either in volcanoes or as extensive sheets building up a lava plateau, which may cover hundreds of thousands of square kilometers and may be fed by numerous fissures¹¹. Previous studies on the North Arabian volcanic province suggest that the derivation of this basalt from deep mantles source material and that it is characterized by a low degree of partial melting of upper mantle peridotites with the minor secondary^{12,13}. The study area is located in the Southern part of Hail region. It is located between 27° 13'N and 27° 31'N and 42° 09'E and 42° 35'E. The principal features of the region are the two great mountain ranges of Aja (granites) and Salma (basalts) and the immense rolling sand dunes of An-Nafūd¹⁴⁻¹⁶.

MATERIALS AND METHODS

Samples preparation and irradiation: A total of 10 samples of basalt rock collected from Hail in Saudi Arabia for investigation by instrumental neutron activation analysis. The powdered samples were sieved using a standard set of sieves to a diameter range of less than 125 mm and greater than 63 mm. Each powdered sample were homogenized using an electric shaker. The samples then irradiation by thermal neutrons at Act labs in Canada. The 4E-Research-INAA, total digestion-ICP, lithium metaborate/tetraborate fusion-ICP is used. A 30 g aliquot is encapsulated in a polyethylene vial and irradiated with flux wires at a thermal neutron flux of $7 \times 10^{12} \text{ n cm}^{-2} \text{ sec}^{-1}$. After a 7-day decay to allow Na-24 to decay the samples are counted on a high purity Ge detector with resolution of better than 1.7 keV for the 1332 keV Co-60 photopeak. Using the flux wires, the decay-corrected activities are compared to a calibration developed from multiple certified international reference materials. Table 1 shows the nuclear data for the elements determined by INAA. Hoffman¹⁷, gives the technical details of INAA such as the radioisotopes used to calculate the concentration of the analyzed elements as well as their nuclear data.

For measuring the elements not detected by INAA like: Be, Cu, Dy, Er, Ga, Ge, Gd, Ho, Nd, Ni, Pb, Sn, Tm, V and Y. A 0.2 g sample is mixed with a mixture of lithium metaborate/lithium tetraborate and fused in a graphite crucible. The molten mixture is poured into a 5% nitric acid solution and shaken until dissolved (~30 min). The samples are run for selected traces on a Varian Vista ICP. A 0.25 g sample is digested with four acids beginning with hydrofluoric, followed by a mixture of nitric and perchloric acids, heated using precise programmer controlled heating in several ramping and holding cycles, which takes the samples to dryness. After dryness is attained, samples are brought back into solution

Table 1: Analytical parameters of determining Au+34 packages by INAA¹⁷

Elements	Measured isotopes	T _{1/2}	Principal energy used (keV)	Detection limit
Au	¹⁹⁸ Au	2.7 d	411.8	2 ppm
Ag	^{110m} Ag	249.8 d	657.8, 884.7, 837.5	5 ppm
As	⁷⁶ As	26.3 h	559.1, 657.1, 1216.1	0.5 ppm
Ba	¹³¹ Ba	11.8 d	216, 373, 496	50 ppm
Br	⁸² Br	35.3 h	554, 777, 619, 1044	0.5 ppm
Ca	⁴⁷ Ca	4.54 d	1297.1	1%
Co	⁶⁰ Co	5.27 y	1173.2, 1332.4	1 ppm
Cr	⁵¹ Cr	27.7 d	320.1	5 ppm
Cs	¹³⁴ Cs	2.06 y	796, 604.7	1 ppm
Fe	⁵⁹ Fe	44.5 d	1099.3	0.01%
Hf	¹⁸¹ Hf	42.4 d	482	1 ppm
Hg	²⁰³ Hg	46.6 d	279.2	1 ppm
Ir	¹⁹² Ir	73.8 d	316.5, 468.1	5 ppm
Mo	^{99m} Tc	65.94 h	140.5, 739.6	1 ppm
Na	²⁴ Na	14.96 h	1368.6	0.01%
Ni	⁵⁸ Co	70.8 h	810.8	20 ppm
Rb	⁸⁶ Rb	18.7 d	1076.6	15 ppm
Sb	¹²² Sb	2.7 d	564.2	0.1 ppm
Sc	⁴⁶ Sc	83.3 d	889, 1120	0.1 ppm
Se	⁷⁵ Se	119.8 d	136, 264.7	3 ppm
Sn	^{117m} Sn	13.6 d	159	0.02 ppm
Sr	⁸⁵ Sr	64.8 d	514	0.05%
Ta	¹⁸² Ta	114.5 d	1121, 1221	0.5 ppm
Th	²³³ Pa	27.4 d	311.8	0.2 ppm
U	²³⁹ Np	2.36 d	106.1, 277.6, 228.2	0.5 ppm
W	¹⁸⁷ W	23.9 h	479, 686	1 ppm
Zn	⁶⁵ Zn	244 d	1115.6	50 ppm
La	¹⁴⁰ La	40.3 h	329, 487, 816, 1596	0.5 ppm
Ce	¹⁴¹ Ce	32.5 d	145.4	3 ppm
Nd	¹⁴⁷ Nd	10.98 d	91, 531	5 ppm
Sm	¹⁵³ Sm	64.3 d	103.2	0.1 ppm
Eu	¹⁵² Eu	13.33 y	121, 344, 964, 1408	0.2 ppm
Tb	¹⁶⁰ Tb	72.3 d	298.6, 879	0.5 ppm
Yb	¹⁷⁵ Yb	4.2 d	282, 396	0.2 ppm
Lu	¹⁷⁷ Lu	6.71 d	208.4	0.05 ppm

d: Days, h: Hours and y: Years

using hydrochloric acid. With this digestion, certain phases may be only partially solubilized. Fused sample is diluted and analyzed by Perkin Elmer Sciex ELAN 6000, 6100 or 9000 ICP/MS.

RESULTS AND DISCUSSION

Fourty elements were investigated and identified in basalt rock by instrumental neutron activation by Au+34 elements package in ACT lab and inductively coupled plasma mass spectrometer. Table 1 show the concentration values of the elements in basalt rock collected from Hail, Saudi Arabia. Twenty five elements were detected and determined by instrumental neutron activation: As, Co, Cr, Sb, Sc, Zn, Ba, Br, Sr, Zr, Cs, Hf, Mo, Rb, Th and U in addition to eight rare earth elements, La, Ce, Nd, Sm, Eu, Yb, Tb and Lu. Ten elements in Au+34 elements package were under the detection limit: Ag, Hg, Ir, Ni, Rb, Se, Sn, Ta, W and Hf. The average concentration values sare expressed in units of ppm for all elements. The

results show relatively lower levels of toxic elements such as As, Sb and Cs etc. in basalt rocks with a lower possibility of environmental impact to the surroundings through their discharge. Table 2 shows the concentration of 15 elements determined by inductively coupled plasma mass spectrometer. The elements determined are Be, Cu, Dy, Er, Ga, Ge, Ge, Ho, Nb, Ni, Pb, Sn, Tm, V and Y. The accuracy can be considered satisfactory being for most elements our results in the range of 310% of the reference values, a good precision has also been obtained in most cases.

The concentrations of rare earth elements: La, Ce, Nd, Sm, Eu, Yb, Lu and Tb were determined using INAA and given in Table 2. The selection of photopeaks for the analysis is briefly discussed below for each element. For lanthanum, the high abundance photopeak of ¹⁴⁰La at 1596 keV was used, which is free of interference. The other peak at 487 keV cannot be used due to interferences from ⁴⁷Ca and ¹⁹²Ir. For cerium, the photopeak of ¹⁴¹Ce at 145 keV was used. For neodymium, the photopeak of ¹⁴⁷Nd at 531 keV that is free of interference was

Table 2: Elemental analysis of basalt by different analytical techniques

Elements	Analysis method	Detection limit (ppm)	Concentration (ppm)
As	INAA	1.0	4
Co	INAA	0.1	41.6
Cr	INAA	0.5	204
Sb	INAA	0.1	4.2
Sc	INAA	0.01	20
Zn	INAA	1.0	79
Ba	INAA	1.0	370
Br	INAA	0.01	9.8
Sr	INAA	2.0	1079
Zr	INAA	1.0	210
Cs	INAA	0.1	0.2
Hf	INAA	0.1	5.3
Mo	INAA	2.0	3
Rb	INAA	1.0	21
Ta	INAA	0.01	5.4
Th	INAA	0.05	3.82
U	INAA	0.10	1.20
La	INAA	0.05	37
Ce	INAA	0.05	76
Nd	INAA	0.05	34.3
Sm	INAA	0.01	17.7
Eu	INAA	0.005	2.35
Tb	INAA	0.01	0.88
Yb	INAA	0.01	203
Lu	INAA	0.002	0.305
Be	FUS-ICP	1.0	2
Cu	TD-ICP	1.0	39
Dy	FUS-MS	0.01	0.09
Er	FUS-MS	0.01	2.39
Ga	FUS-MS	1.0	19
Ge	FUS-MS	0.5	1.3
Gd	FUS-MS	0.01	6.29
Ho	FUS-MS	0.01	0.09
Nb	FUS-MS	0.2	80
Ni	TD-ICP	1.0	84
Pb	TD-ICP	5.0	477
Sn	FUS-MS	1.0	2.6
Tm	FUS-MS	0.005	0.319
V	FUS-ICP	5.0	213
Y	FUS-ICP	1.0	20

used. Other peaks that cannot be resolved from the peak of ^{177m}Lu surround the high abundance peak at 91 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 and ^{233}Pa . For lutetium, the results are based on the total peak with a correction for the ^{239}Np presence based on another ^{239}Np peak^{18,19}.

In Neutron Activation Analysis (NAA), the following three types of interfering nuclear reactions should be taken into account²⁰. (1) Nuclides produced by (n, p) and (n, α) reactions on the other heavier elements, which are often the same as the nuclide produced by the (n, γ) reactions. (2) Fission products of uranium, which are the same as the radionuclides for the determination of some REEs, zirconium, ruthenium and molybdenum. (3) Daughter nuclides which succeed the (n, γ) reactions of different target elements and become the same as radionuclides of interest. The isotopes ^{140}La , ^{141}Ce and ^{147}Nd commonly used in the activation analysis of the corresponding elements are also produced by fission of ^{235}U ²¹.

The elemental concentration of uranium from ^{238}U and thorium from ^{232}Th in basalt have been also determined in basalt samples collected from Hail, Northeast of Saudi Arabia (Table 2). The activation converts ^{238}U and ^{232}Th into ^{239}Np and ^{233}Pa , respectively, by neutron capture and successive β-decay^{22,23} given by Eq. 1 and 2:

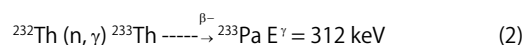


Table 2 shows that the average concentrations values of uranium and thorium obtained in this study are 1.2 and 3.82 ppm, respectively. These values lower than the concentration of uranium and thorium in upper earth crust 1.7 and 8.5 ppm²⁴.

CONCLUSION

It is clear that the NAA technique can be used as a powerful tool to obtain accurate concentration values of basalt rock samples. Twenty five elements were determined by instrumental neutron activation: The elements determined are: As, Co, Cr, Sb, Sc, Zn, Ba, Br, Sr, Zr, Cs, Hf, Mo, Rb, Th and U in addition to eight rare earth elements, La, Ce, Nd, Sm, Eu, Yb, Tb and Lu. In addition, 15 elements were determined by inductively coupled plasma mass spectrometer. The elements determined are: Be, Cu, Dy, Er, Ga, Ge, Ho, Nb, Ni, Pb, Sn, Tm, V and Y. It is hoped that the data presented here will be useful to those dealing with geochemistry of basalt rock and related fields.

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