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Neutron activation analysis of ceramic tiles and its component and radon exhalation rate

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Abstract: The concentrations of 20 trace elements in several ceramics tiles and ceramic composites used in Egypt were elementally analyzed by neutron activation analysis(NAA) technique. The samples and standard were irradiated with reactor for 4 h(in the Second Research Egyptian Reactor(Et-RR-2)) with thermal neutron flux 5.9×10^{13} n/(cm^2·s).

The gamma-ray spectra obtained were measured for several times by means of the hyper pure germanium detection system(HPGe). Also a solid state nuclear track detector(SSNTD) CR-39, was used to measure the emanation rate of radon for these samples. The radium concentrations were found to vary from 0.39—3.59 ppm and the emanation rates were found to vary from $(0.728—5.688) \times 10^{-4}$ kg/(m^2·s).

The elemental analysis of the ceramic tiles and ceramic composites have a great importance in assigning the physical properties and in turn the quality of the material.

Keywords: ceramic; neutron activation analysis(NAA); radon emanation; solid state nuclear track detector(SSNTD)

Introduction

Ceramic, consist of earthy materials like clay kaolin, lime stone and sand glass. Ceramic materials are non metalic, inorganic compounds primary oxides, also carbides, nitrides and silicides. Two types of bonding mechanisms occur in ceramic material, ionic and covalent. The over all properties of these materials depend on the dominant bonding mechanism. Knowing the elemental analysis of ceramics, and its bonding, can be used in different applications including high heat capacity and low heat conductance, corrosion resistance, electrical insulation, semiconductance, or superconductance, nonmagnetic and magnetic, hard and strong(Hopkins, 1985; Rovner, 1976; Anderson, 1995).

It was necessary to obtain information on a number of major, minor and trace elements within relatively short times, as the analysis of a large number of samples was needed.

Non-destructive analytical methods were favoured, as the application of digestion or leaching procedures during the sample preparation could cause losses of some of the elements(Karanatsios, 1988). The NAA characteristics of pure ceramics make them very attractive for application in decorest of houses. The activation analysis technique has been used to study in alloys(Eissa, 1998), fertilizer (Abdel-Haleem, 1997), soil samples and plant(Smodis, 1995), and rocks samples(Spiridon, 1995).

During the last years a research project concerning multielement analysis of natural gelogical samples used in fabricate the ceramic tiles by means of instrumental NAA required a suitable reference material for testing the applicability of the analytical procedure to gelogical materials.

For analysis the so-called absolute monostandard method was used, i.e. the element contents were not determined by comparison with standard refercence material SRM's but by calculating the absolute concentration from the measured γ-activity. For this purpose the measuring devices have to be calibrated on an absolute scale with radioactive standard specimens. Furthermore, a precise knowledge of the irradiation and counting parameters(irradiation and decay period, sample-detector distance, counting period), and the nuclear data of the radionuclides(decay scheme, decay constant, nuclear reaction cross-sections, fractional isotopic abundance of the parent isotopes, etc.) is necessary for calculating the absolute element contents.

Survey had been carried out for a selected samples of ceramic tiles to provide a usable picture of environmental gamma background radiation. The requirement for the determination of the radio elements has become a matter of interest in public health due to their hazardous nature with respect to internal exposure. Emanation of radon(^{222}Rn), an α-radioactive inert gas, is associated with the presence of radium and its ultimate precursor uranium in the ground. The inhalation of its short-lived daughter products is a major contributor to the total radiation dose to exposed subjects. In the uranium mining environment, large dose due to radon progenies may be sufficiently high to cause an increase in occurrence of lung cancer(UNSCEAR, 1993). The use of solid state nuclear track detectors is a convenient technique for low activity measurements since it is low cost, simple operation, high registration sensitivity, and possiblity of using for long period of exposure without any fading.

The aim of the present work is to establish a fast, non-destructive and accurate method to determine the majour, minor and trace elements in some types of ceramic for wall and floor and some of the composites used in fabrication of ceramic tiles from the locally produced in Egypt, by utilizing secound research reactor and detecting the gamma ray lines by a HPGe detection system. Also, in the present work, we give an estimation for the radon emanation using selective groups of the studied samples.

1 Experimental work

1.1 Sample preparation

Six samples of the composite of ceramic tiles were obtained from

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various sources in the local market in Egypt, sample 1 (red feldspar), sample 2 (Aswan clay), sample 3 (kaolin), sample 4 (lime stone), sample 5 (white feldspar) and sample 6 (sand glass). Also, two samples of fabrication ceramic tiles used in the floor, A (El-Gawhara) and B (Clupatra). Also three sample of fabrication ceramic tiles used in the wall C (El-Gawhara), D (Maser) and E (El-Ahaliua). Five samples from each type are obtained for accuracy.

The samples were ground, sieved at 200 mesh and prepared for neutron activation analysis (NAA). A 100 mg from each sample was sealed in clean aluminum foil (Sroor, 2000), used as a container for irradiating the sample. An empty aluminum foil was used for background assessment. A standard reference material (IAEA, soil 7) (IAEA, 1991) was used for a relative calculation and quality accuracy. A gold monitor sample was used to determine the thermal neutron flux. All the samples and the standard reference material were irradiated for four hours at an average thermal neutron flux 5.9×10^{13} n/(cm^2 · s) in the Second Research Egyptian Reactor (Et-RR-2).

Preparation of the samples for radon emanation were carried out by drying the sample in an oven at a temperature of 110℃. Each sample was placed in a glass cylinder of radius 3.5 cm and 10 cm length. Dosimeters were prepared by putting two CR-39 detectors in the inner surface of the chamber cover. The cylindrical container was sealed, the samples were stored for at least 30 d to reach secular equilibrium.

1.2 Proceding

In the γ-spectroscopy measurements the single spectra were collected in optimum fixed geometry using hyper pure germanium (HPGe) coaxial detector coupled to a computerized multichannel analyzer through a high sensitive electronic system. The detector has a resolution of 1.79 keV full width at half maximum (FWHM) at 1332.5 photopeak of ^{60}Co, with an efficiency of 15% and a peak to composition ratio of 55:1. The samples were left to cool for about one week. The distance between sample and detector was 10 cm. A gamma-ray spectrum of each irradiated sample was collected for 2 h and repeated weekly for one month. The detector and the attached electronic circuits in conjunction with the computerized multichannel analyzer were used for measuring the induced radioactivity. Energy and efficiency calibration up to about 3 MeV was carried out using the multi-gamma ray standard source (MGS-4) (NMC, 1998).

The radon emanation rate can be made using CR-39 detectors because of their capability to register tracks at different levels of registration sensitivity. The calibration of CR-39 should be performed where the integrating radon's concentration is known (Khan, 1990). After 30 d the films were then exposed to radon and its daughters in the chamber for a known period of time. The exposure of the detectors, followed by etching of the tracks left in the film provided the concentration of radon and its daughters. After etching, the detector was washed in bi-distilled water, dipped for a few seconds in 3% acetic acid solution, washed again and allowed to dry in air. The track density per m^3 (Khan, 1990) was determine using an optical microscope.

1.3 Theoritical background for radon emanation

Radon is an inert gas ($T_{1/2}$ = 3.825 d), it can be traverse in soil and other materials radon emanation is the fraction of the radon atoms which released from the rock to the surrounding phases. There are saveral parameters that affect the radon release rate such as: (1) the radon concentration; (2) the porosity and permeability of the material; (3) the nature of the surrounding medium.

Radon emanation (E_m) rate relative to real radium content can be calculated from the relation:

$$E_m = \frac{\text{The radon exhalation rate}}{\text{The radium concentration}}.$$

The exhalation rate of radon (Khan, 1990) can be calculated from the relation:

$$E_x = \frac{P v \lambda}{\zeta A T_{eff}},$$

where E_x is the radon exhalation rate (Bq/(m^2 · s)), p is the track density as measured by CR-39 detector, T/m^2, v is the effective volume of the cylindrical container (m^3), λ is the radon decay constant, ζ is the detector efficiency, A is the area of the cylinder chamber and T_{eff} is the effective exposure time = $T + 1/\lambda\,(e^{-\lambda T} - 1)$, T is the exposure time.

2 Results and discussion

To confirm the accuracy of the detection system, the results of some elements which are well resolved for the standared reference material (SRM) soil-7 are compared with the certified values in Table 1. Although the absolute INAA does not need standard reference materials, it is practical to confirm the procedure, especially when a new matrix is to be investigated. The results given in Table 1 show good agreement with the certified values.

Table 1 Analysis of the standard reference material IAEA-soil 7

Element	Present work, ppm	Certified, ppm	Ratio
Co	8.07 ± 0.4	8.9	0.91
Cr	57.56 ± 2.88	60	0.96
Cs	5.14 ± 0.25	5.4	0.95
Eu	0.97 ± 0.04	1	0.97
Hf	5.16 ± 0.25	5.1	1.01
La	27.11 ± 1.35	28	0.97
Sc	8.5 ± 0.42	8.3	1.02
Sm	5.04 ± 0.25	5.1	0.99

The qualitative NAA measurements on ceramic tiles and its component samples led to the detection of the major element (Fe), the minor element (Cr, Co, Zn, Ba, Ce, and Ta) and the trace elements (Sc, La, Eu, Sm, Ca, Tb, Yb, Lu, Hf, Th and U). These elements were identified by interpretation of γ-ray singles spectral scans such as that in Fig.1 (a-b), shows a partial gamma-ray singles spectra in the range 200—900 keV for kaolin and glass sand samples, also Fig.2 (a-b), shows a partial gamma-ray singles spectra in the range 200—900 keV for ceramic El-Gawhara tiles for wall and floor respectively.

The quantitative of the γ-ray spectra analysis for elements over limited concentration ranges were based on the construction of a calibration and efficiency curves. The concentration values of the major, minor and trace elements presented in these samples have a great importance in assigning the physical properties (elasticity, homogeneity, insulation properties, stability and mechanical properties) and in turn the quality of the material.

Table 2 shows the elements which were positively identified using INAA technique for the ceramic composites. From Table 2, we can see that the sample 1 (red feldspar) contains Zn with 668.71 ppm, while sample 5 (white feldspar) not contains Zn. Also samples 1 and 5 contain U(^{239}Np) with the same value ~ 2.6 ppm. It is shown that from this table, the sample 2 (Aswan clay) contains the lowest elements with

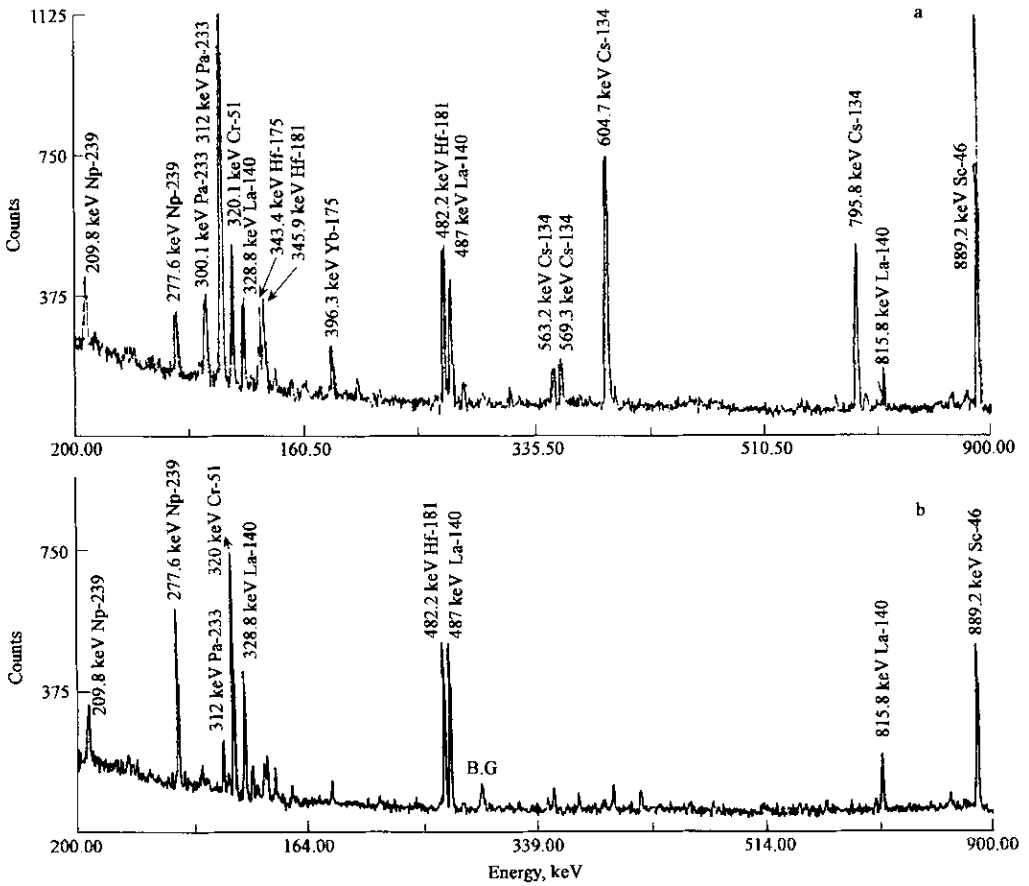

Fig. 1 Portion of the gamma-ray singles spectrum in the range between 200—900 keV for kaolin and glass sand samples

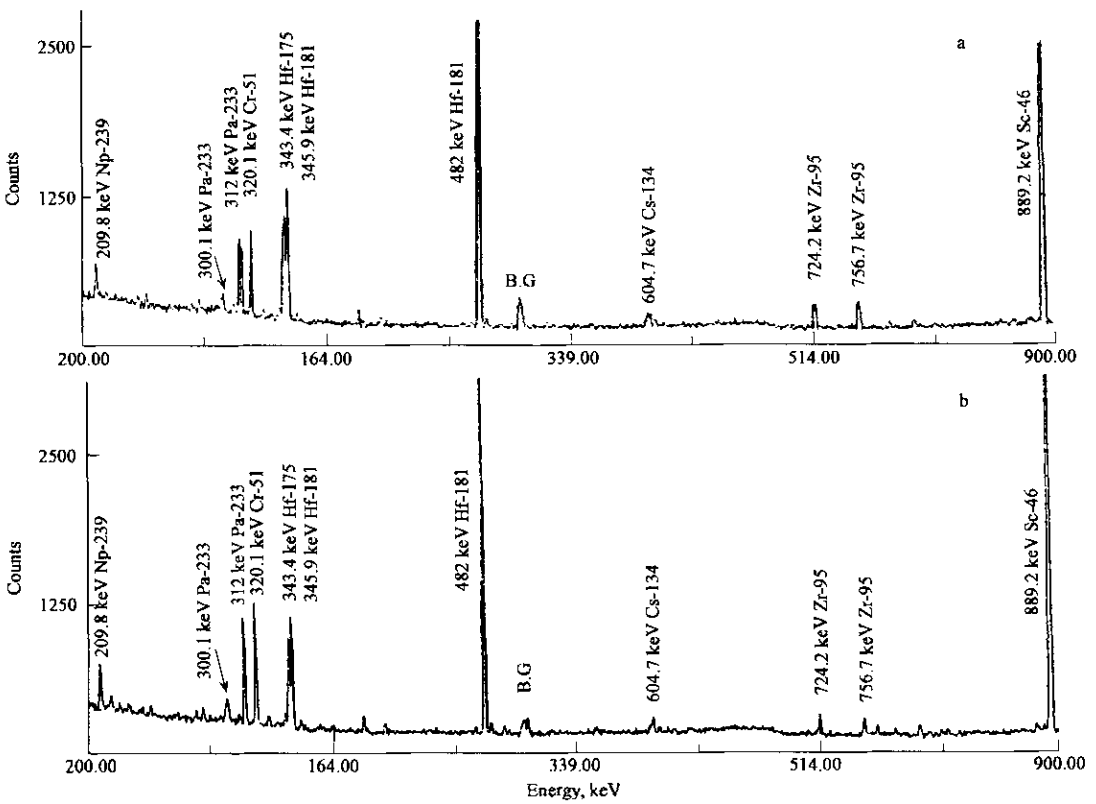

Fig. 2 Portion of the gamma-ray singles spectrum in the range between 200—900 keV for ceramic El-Gawhare tiles for wall and floor

highest concentration for the elements (Sc, Cr, Co, Ce and Eu). Sample 3(kaolin) contains 18 elements within the range 0.64 to 14761.9 ppm. Lime stone(sample 4) contains 16 elements within the range 0.31 to 18171.9 ppm.

Table 2 Concentration of the major, minor and trace elements(in ppm) for the component of ceramic tilles

Element	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
Sc-46	1.34 ± 0.07	31.05 ± 1.56	3.19 ± 0.15	0.84 ± 0.042	1.21 ± 0.06	0.25 ± 0.01
Cr-51	96.15 ± 4.81	155.15 ± 7.75	13.8 ± 0.39	33.02 ± 1.65	23.03 ± 1.15	33.55 ± 1.67
Fe-59	3688.6 ± 2684	130755 ± 6537	14761.9 ± 738.1	18171.9 ± 908.5	11123.1 ± 556.1	16278.9 ± 813.5
Co-60	10.38 ± 0.52	36.05 ± 1.81	2.38 ± 0.11	4.56 ± 0.23	1.45 ± 0.07	3.98 ± 0.19
Zn-65	668.71 ± 33.43	–	–	144.69 ± 7.2	–	140.14 ± 7.01
Ba-131	–	–	450.5 ± 22.5	–	163.3 ± 8.16	
Cs-134	–	8.25 ± 0.4	32.3 ± 1.61	1.72 ± 0.08	–	1.36 ± 0.06
La-140	4.15 ± 0.21	–	21.67 ± 1.08	4.22 ± 0.21	20.99 ± 1.04	3.25 ± 0.16
Ce-141	3.51 ± 0.18	93.15 ± 4.65	55.86 ± 2.79	14.52 ± 0.73	16.8 ± 0.8	6.21 ± 0.31
Eu-152	0.59 ± 0.03	2.17 ± 0.10	0.95 ± 0.04	0.38 ± 0.02	1.01 ± 0.05	0.39 ± 0.01
Sm-153	0.29 ± 0.02	–	5.03 ± 0.25	0.67 ± 0.03	5.01 ± 0.25	0.51 ± 0.02
Tb-160	–	1.51 ± 0.07	0.64 ± 0.03	0.31 ± 0.01	3.54 ± 0.17	–
Yb-169	–	3.38 ± 0.16	2.25 ± 0.11	–	6.91 ± 0.34	–
Yb-175	–	–	1.91 ± 0.09	0.42 ± 0.02	9.61 ± 0.5	1.16 ± 0.05
Lu-177	–	–	0.82 ± 0.04	–	1.97 ± 0.09	–
Hf-181	8.16 ± 0.41	8.44 ± 0.42	4.63 ± 0.23	2.85 ± 0.14	8.73 ± 0.43	4.88 ± 0.24
Ta-182	63.8 ± 3.19	2.12 ± 0.11	4.38 ± 0.21	74.06 ± 3.71	2.19 ± 0.11	3.06 ± 0.15
Pa-233	0.97 ± 0.04	1.17 ± 0.06	1.58 ± 0.08	1.07 ± 0.05	1.92 ± 0.09	0.94 ± 0.02
Np-239	2.65 ± 0.13	1.64 ± 0.08	2.96 ± 0.15	1.34 ± 0.07	2.61 ± 0.13	3.59 ± 0.18

Table 3 shows the comparison between the present work of sample 6 (sand glass) and the certified of UNS-SPS glass sand(Gerler, 1991) with the measured value in the study of J. Gerler *et al*. From this comparison, we can see that good agreement between the present work and those for the elements Sc, La, Ce and Th.

Table 3 Comparisoin between the concentration of the UNS-SPS glass sand certified reference value, study of J.Gerler *et al*., and the glass sand of the present work

Element	Certified reference, Gerler, 1991	J.Gerler *et al*., 1991	Present work
Sc	0.27	0.28	0.25
Co	0.48	0.47	3.98
La	2.42	3	3.25
Ce	6	6	6.21
Eu	0.07	0.06	0.39
Th	1.04	1.17	0.94

Table 4 shows the concentration elements for the sample of fabriation ceramic tiles used in the floor(A and B). The elements Fe, Zn and Ba contain large concentration and the two sample contain nearly the same concentration(2.03—2.56 ppm) for ^{232}Th indicated by ^{233}Pa, while sample A contains U with concentration 1.44 ppm and sample B contains U with 1.56 ppm. The two samples A and B contain the element of Zr with the concentration 0.4 and 1.13 ppm respectiviely. The Zr element introduced in the composite of glaze coated the surface of the ceramic tile.

Table 5 shows the concentration of elements for some types of ceramic tiles used in the wall. Sample C was not contains the element of Ba and the concentration of the other element were between 0.53 to 30465.6 ppm. Sample D, not contains the elements La and Sm. The highest element concentration was Fe(51596.2 ppm), while the lowest element concentration was U(Np^{239}) ~ 0.21 ppm. Sample E, contains twenty elements with the concentration between 0.31 to 51846.9 ppm. The concentration of Fe in ceramic tiles used in the floor is higher than the ceramic tiles used in the wall which increasing the hardness of the tiles. Also the concentration of U and Th in the ceramic tiles for floor or wall were in the permissible level.

The alpha-activity of the radionuclides in ceramic samples can be related to the number of tracks registered in plastic detectors during certain exposure time. In order to evaluate the radon risk in a given atmosphere, it is necessary to check the relation between the activity concentrations of uranium and radon for some ceramic samples. In the present work, solid state nuclear track detectors technique have been applied, CR-39, for the emanation rate measurements.

Table 4 Concentration of elements(in ppm) for different kinds of tiles ceramic used in the floor

Element	Sample A	Sample B
Sc-46	15.17 ± 0.75	16.61 ± 0.83
Cr-51	100.53 ± 5.02	92.70 ± 4.63
Fe-59	48164.7 ± 2408.1	56967.3 ± 2848.3
Co-60	15.45 ± 0.77	9.05 ± 0.45
Zn-65	1888.5 ± 94.42	6025.18 ± 301.25
Zr-95	0.4 ± 0.02	1.13 ± 0.05
Ba-131	761.57 ± 38.07	1942.66 ± 97.13
Cs-134	5.24 ± 0.26	4.55 ± 0.22
La-140	27.70 ± 1.38	–
Ce-141	56.69 ± 2.83	47.03 ± 2.35
Eu-152	1.02 ± 0.05	1.60 ± 0.08
Sm-153	5.41 ± 0.27	4.65 ± 0.23
Tb-160	1.50 ± 0.07	0.49 ± 0.02
Yb-169	3.96 ± 0.19	5.45 ± 0.27
Yb-175	4.40 ± 0.22	3.61 ± 0.18
Lu-177	1.37 ± 0.06	126.9 ± 6.34
Hf-181	53.22 ± 2.66	165.35 ± 8.26
Ta-182	2.30 ± 0.11	3.20 ± 0.16
Pa-233	2.03 ± 0.10	2.56 ± 0.13
Np-239	1.44 ± 0.07	1.56 ± 0.08

Table 5 Concentration of elements(in ppm) for different kinds of ceramic tiles used in the wall

Element	Sample C	Sample D	Sample E
Sc-46	12.20 ± 0.61	16.94 ± 0.84	13.95 ± 0.69
Cr-51	73.23 ± 3.61	176.4 ± 8.82	145.53 ± 7.27
Fe-59	30465.6 ± 1523.2	51596.2 ± 2579.1	51846.9 ± 2592.3
Co-60	13.41 ± 0.67	25.97 ± 1.29	20.92 ± 1.04
Zn-65	5362.28 ± 268.1	4839.39 ± 241.86	2067.68 ± 103.3
Zr-95	0.53 ± 0.02	0.56 ± 0.002	0.31 ± 0.01
Ba-131	-	823.30 ± 41.16	2027.66 ± 104.8
Cs-134	4.24 ± 0.21	3.48 ± 0.17	2.44 ± 0.12
La-140	41.81 ± 2.09	-	41.85 ± 2.09
Ce-141	54.21 ± 2.71	56.20 ± 2.81	50.71 ± 2.53
Eu-152	1.15 ± 0.05	1.18 ± 0.05	1.11 ± 0.06
Sm-153	4.15 ± 0.20	-	4.16 ± 0.20
Tb-160	1.41 ± 0.07	1.19 ± 0.05	6.23 ± 0.31
Yb-169	4.01 ± 0.20	4.03 ± 0.20	4.15 ± 0.20
Yb-175	2.16 ± 0.10	2.21 ± 0.11	4.81 ± 0.24
Lu-177	1.26 ± 0.06	2.36 ± 0.11	1.32 ± 0.06
Hf-181	71.24 ± 3.56	73.82 ± 3.69	41.67 ± 2.08
Ta-182	2.83 ± 0.14	3.51 ± 0.17	0.81 ± 0.04
Pa-233	1.99 ± 0.09	2.82 ± 0.14	3.11 ± 0.16
Np-239	1.92 ± 0.09	0.21 ± 0.01	0.39 ± 0.02

The main sources of radon is the soil, ground water and building material which considerd as a natural radon sources. These sources were manifested by its emanation power, effective and real radium content. Emanation of radon is associated with the presence of radium and its ultimate precursor uranium in the ground. Measurement of ^{226}Ra were carried out by gamma-spectroscopy, the calculated decay rate of ^{222}Rn emanated from each sample include correction for equilibration time.

Ceramic was found to emanate ^{222}Rn to produce an average of $(3.72 \pm 0.09) \times 10^{-4}$ kg/(m^2·s). Comparison of ^{222}Rn emanation to ^{226}Ra indicates emanation < 0.059 % of the consistent with the presence of ^{222}Rn progeny.

Zeman and Hon(Zeman, 1994) reported the high radon emanation from glazed ceramics. They imparted gamma-spectrosopy data as indicated a substantial disequilibrium between progeny of ^{238}U before and after radon emanation. These studies showed that uranium compounds may emanate measurable ^{222}Rn. They suggested that uranium glazed ceramics can be a significant contain ^{222}Rn. Table 6 represents the present results, the radon emanation were from 0.728×10^{-4} to 5.688×10^{-4} kg/(m^2·s), the large variation may be attributed to the variation of uranium concentrations according to the composite of ceramic. The values of the radon emanation rate from the ceramic samples are found to correspond with the values of uranium concentration measured by hyper pure germanium detector in the corresponding sample.

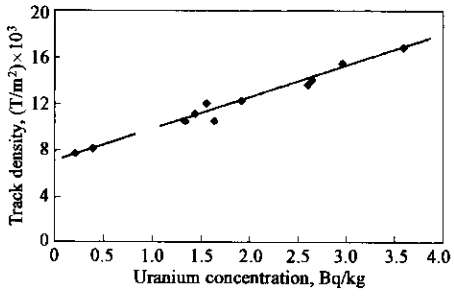

Fig.3 Relation between the activity concentration of uranium and the track density

Table 6 The track density, redium cncentration and emanation rate of radon

Sample	Radium concentration, × 0.08 Bq/kg	Track density, × 10^3 T/cm^2	Emanation rate, × 10^{-4} kg/(m^2·s)
1	2.65 ± 0.10	13.99 ± 0.45	5.05 ± 0.05
2	1.64 ± 0.08	10.47 ± 0.73	3.78 ± 0.06
3	2.96 ± 0.15	15.44 ± 0.89	5.11 ± 0.05
4	1.34 ± 0.07	10.45 ± 1.13	3.41 ± 0.11
5	2.61 ± 0.13	13.61 ± 1.10	4.50 ± 0.05
6	3.59 ± 0.18	16.75 ± 1.32	5.69 ± 0.08
A	1.44 ± 0.07	11.12 ± 1.04	3.46 ± 0.12
B	1.56 ± 0.08	12.01 ± 0.81	3.74 ± 0.13
C	1.92 ± 0.09	12.25 ± 0.86	4.17 ± 0.07
D	0.21 ± 0.01	7.71 ± 1.13	0.73 ± 0.14
E	0.39 ± 0.02	8.10 ± 1.23	1.28 ± 0.15

From Fig.3, it is evident that there are a linear relation between the activity concentration of uranium and the radon track density.

3 Conclusions

From the results presented it can be concluded that neutron activation analysis is optimized method in industrial applications, such as for analysis the powders used in the production of ceramics and also the ceramic tiles for floor and wall. The concentration values of the major, minor and trace elements presented in this samples have a great importance in assigning the physical properties and in turn the quality of the material.

It is hoped that this work can give some new information about the trace elements constitution of the investigated ceramic to establish their concentration levels with respect to the decoration and environmental points of view.

Also from the results, an average radon emanation from ceramics produce of $(3.666 \pm 0.096) \times 10^{-4}$ kg/(m^2·s). Emanation level for individual ceramics may vary significantly from the average value because of differences in ceramic materials and emanation efficiencies. The values of radon emanation rate from ceramic samples are found to correspond with the measured values of uranium in the corresponding samples. The applied solid nuclear track detectors, CR-39 provides a simple method to measure the actual radon emanation rate in the laboratory.

A combined technique of nuclear track density counting and gamma spectroscopic analysis has proved to be an adequate and reliable technique for radioactivity determination.

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