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Elemental Analysis of Raw Materials of Nuclear Reactor Shielding to Develop Low Activation Concrete

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ABSTRACT

In this study, elemental concentrations of Ce, Hf, Fe, Sb, Tb, Sc, Ta, Zn, Cs, Co and Eu in raw materials of reactor biological shielding (cement, sand and heavy minerals separated from beach sand) are determined by research reactor-based instrumental neutron activation analysis technique (INAA) so that a strategy can be made to develop low-activation concrete. These elements are mainly responsible of long-lived radionuclides induced in biological shielding of a reactor during its operation. The concentrations of the studied elements in white Portland cements are much lower in comparison with those in ordinary Portland cements. This study reveals that inland sands contain low concentrations of the studied elements than those of beach sands. Elemental compositional data of the shielding materials can be effectively used to choose ingredients necessary for constructing radiation shielding of a nuclear installation to reduce radiation hazard.

Keywords: Elemental composition, Shielding ingredients, Residual activity, Low-activation concrete, Instrumental Neutron Activation Analysis

1. Introduction

Due to high level of neutron and gamma doses, decommissioning of nuclear facilities is associated with particular technical challenges and risks related to human health and the environment. Worldwide there are large numbers of nuclear power stations and accelerators producing radioactive waste. Some of them are coming to the end of their operating lives and will require decommissioning. After decommissioning a nuclear reactor, long-lived radioisotopes formed by (n,γ) reaction in biological concrete shielding cause difficulty in waste management of the activated shielding [1,2]. Therefore, decommissioning of nuclear installations after their operating life-time and their dismantling are connected with the necessity of the disassembling, handling and disposing of a large amount of radioactive equipment and structures. It raises questions about residual radioactivity present in the reactor shielding material. Moreover, activated shielding increases radiation exposure to the maintenance group during fuel recycling and maintenance of the reactor [3,4]. Therefore, elemental analysis of the raw materials of the shielding materials is very important before installing any nuclear facility. Long-lived radionuclides like ^{152}Eu, ^{154}Eu, ^{60}Co, ^{134}Cs etc. are of great concern in the disposal of shielding materials used in nuclear reactor. Several calculations and experimental studies have been done to study the effect of neutron activation on the concrete shielding during the investment or after decommissioning of a

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nuclear facility [5-8]. The radioactive isotopes due to the major elements such as Al, Ca, Na, Mg and Si will not be taken into account because after a short interval of time they will not be effective for residual activity [9, 10].

Concrete is commonly used as a radiation shielding material as it is cheaper, easily molded into complex shape, has good structure and is suitable for neutron and proton shielding compared to other shielding materials [11]. Generally, cement and sand are the main composite materials of concrete, which contain many elements with long-lived nuclei. Bangladeshi cement industry mainly produces Portland composite cement which is a fine, typically gray powder produced by grinding Portland cement clinker (more than 90%), containing maximum about 5% gypsum, which controls the set time, and up to 5% minor constituents. The minor constituent of cement are specially selected from some materials such as blast furnace slag, pozzolanas, silica fume, fly ash, burnt shale and limestone in a proportion exceeding 5% by mass related to the sum of all main and minor additional constituents.

Elemental compositions of sand differ in different sand deposits due to their geological and geographical differences [12]. Sometimes heavy minerals separated from beach sand are also used in concrete to increase density of the heavy concrete up to a required level [13, 14]. Addition of heavy minerals into shielding concrete incorporates trace elements with long-lived radionuclides [15-18]. Therefore, heavy minerals with low elemental contents of interest are also important to develop low activation concrete.

The kind and quantity of shielding material fluctuate according to radiation type, the activity of source and the dose rate. However, there are other factors for choice of shielding materials such as their fabrication, cost and weight [19]. Also, it is very crucial that materials used for this purpose are available in the country. In this respect, it is desirable to have the knowledge about the effective materials for neutron and gamma-ray shielding. Many new countries, both developed and developing, plan to introduce nuclear power plant (NPP). Recently, Bangladesh government has signed an agreement with Russia to install a nuclear power reactor (2 x1000 MW) at Rooppur, Pabna. The biological concrete shielding of the proposed Rooppur NPP is expected to be constructed using local raw materials of shielding. Therefore, it is very important to screen the local raw materials of shielding to develop low activation concrete.

The amount of activation for structural material surrounding a reactor core can easily be calculated when both ingredients of material and the history of neutron irradiation are known. However, most of the long-lived radioisotopes are generated from elements which have very small concentrations may be parts per million (ppm). To obtain precise concentration for these elements in the biological shielding of the reactor by chemical methods is usually difficult. Therefore, instrumental neutron activation analysis (INAA) technique is an accurate method to obtain concentrations of the elements responsible of long-lived induced radioactivity in the reactor shielding [10, 20-23]. Moreover, this technique inmates the real situation of activation of the sample in a nuclear reactor. INAA combined with high-resolution germanium gamma-ray spectrometry has proved to be a powerful multi-element analytical technique for trace contents in different sample matrices.

To reduce residual activity in shielding waste as low as possible, it is necessary to choose concrete compositions with low concentrations of the elements responsible of long-lived radioisotopes. In this study, elemental concentrations of the trace elements responsible of long-lived radioisotopes in Bangladeshi shielding ingredients are determined to develop low-activation concrete.

2. Experimental

2.1 Sample collection and preparation

For this study, six brands of ordinary Portland cements (OPC) (coded as OPC1 to OPC6) and three brands of white Portland cements (WPC) (coded as WPC1 to WPC3) were collected from local markets which are widely used in Bangladesh. Five sand samples from inland deposits of Bangladesh (coded as S1 to S5) which are locally used as building materials and three beach sand samples rich of heavy minerals from Cox's Bazar sea beach (coded as S6 to S8) were collected for this study. The samples of heavy minerals: Zircon, Illmenite and Magnetite were collected from the Beach Sand Minerals Exploitation Center (BSMEC), Cox's Bazar, Bangladesh. In BSMEC, heavy minerals are separated from bulk sand samples collected from Cox's Bazar sea beach. All collected sand samples were purified by removing all contaminants such as rocks, woods, vegetation residual parts etc. Sand and heavy mineral samples were grounded into powder using an agate mortar. Each sample was put into a clean container and dried in an oven until attaining a constant weight. For elemental analysis by INAA, about 60 mg of each sample and standard (certified reference materials (CRM) Soil-7 and SL-1 from International Atomic Energy Agency (IAEA) and standard reference material (SRM) 1633b from National Institute of Standards and Technology (NIST)) was doubly packed separately using clean polyethylene vial and heat sealed. The samples and

standards were put into an irradiation vial along with neutron flux monitor, Au-Al alloy (0.1% Au) at the bottom, middle and top of the stack of the samples.

2.2 Measurement and analysis

The samples and reference standards were irradiated simultaneously for 8 minutes at the rabbit irradiation facility of 3 MW TRIGA Mark-II research reactor (thermal neutron flux: 2.0 x 10^{13} $cm^{-2}.s^{-1}$) at the Atomic Energy Research Establishment, Dhaka, Bangladesh. All the samples and standards were packed tightly (2-3 cm) in a polyethylene irradiation vial and irradiated assuming that the neutron flux had been the same for both of the samples and standards. During irradiation radioactive nuclei are formed from the constituting elements thus allowing their determination by gamma-ray spectrometry. Outer polyethylene pack of each sample and standard was replaced by a new one to avoid radiation hazard. Then they were kept inside lead shielding for one month to decay short-lived radionuclides. The measurement of each sample and standard was carried out for 7200 s with a gamma-ray spectrometry system consisting of Canberra HPGe detector (40% relative efficiency; energy resolution of 1.9 keV at 1332.5 keV energy for Co-60 source) coupled with a PC-based multichannel analyzer (MCA) along with Genie software installed in the PC. The gamma-ray peak analysis was performed using the software Hypermet PC v.5.12. The nuclear data used for elemental analysis by INAA are given in Table 1.

3. Results and discussion

The concentrations of the studied elements responsible for forming long-lived radionuclides in the investigated shielding materials are determined by INAA technique. The studied elements are Ce, Hf, Fe, Sb, Tb, Sc, Ta, Zn, Cs, Co and Eu which have radionuclides with long half-lives (more than one month) as shown in Table 1. These elements are mainly responsible for producing long term induced radioactivity in the reactor shielding after decommissioning of a nuclear reactor. A partial gamma-ray spectrum for neutron irradiation of an ordinary Portland cement sample is shown in Fig. 1. The quality of the data is evaluated by determining the elemental contents of IAEA-CRM-SL-1 and NIST-SRM-1633b relative to IAEA-CRM-Soil-7. In this study, concentrations of the studied elements determined in the reference materials are within 12% deviation from their certified or literature values which ensures the quality of the analysis. In this study, uncertainty associated with the individual concentration value is due to counting statistics (1σ). However, there is commonly 3% more uncertainty, estimated in our analysis, which is also associated with the reported values which include sample and standard preparation (0.55%), irradiation (0.12%), positioning in the detector (2.5%), pulse-pileup losses (0.56) and peak integration (0.38%).

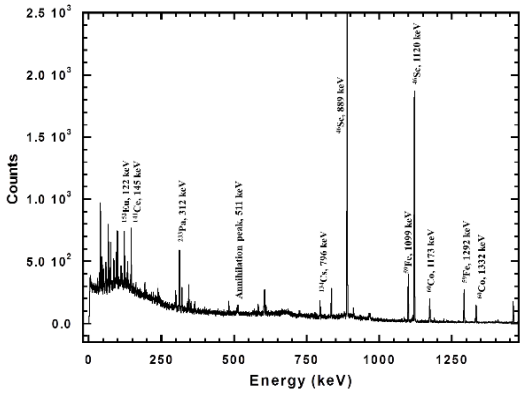

Fig.1 *Partial gamma-ray spectrum of an irradiated ordinary Portland cement sample.*

The concentrations of the studied elements in Bangladeshi cement samples are given in Tables 2 and 3. Among the ordinary Portland composite cements, OP6 cement contains relatively small concentrations of most of the studied elements. It is mentioned in the specification of this cement that this is fly ash free. Since fly ash and blast furnace slug contains high amounts of the studied elements [25], lower concentrations of the studied elements may be due to the fly ash free ingredients of this cement. Therefore, fly ash free cements are more suitable for the biological shielding concrete of a nuclear reactor, because they contain low amounts of the elements responsible for long-lived induced radioactivity in shielding concrete. Average concentrations of the studied elements in WPC are compared with those in OPC in Fig.2. WPC contains lower concentrations of the elements than those in OPC. In case of WPC, specially selected raw materials (like kaolinite and white chalk) are used in cement clinkers which contain low contents of the studied elements. Cost of WPC is higher than those of OPC otherwise WPC could have been suggested for the preparation of concretes for the biological shielding of a nuclear reactor.

Since elemental contents of sand may differ in different sand deposits due to their geological and geographical differences, sands from different Bangladeshi sand deposits are analyzed in this study. The concentrations of the studied elements in sand samples are given in Table 4. It is observed that elemental contents are widely varied in different deposits. When average concentrations of the studied elements in sands from inland deposits (S1 to S5) and beach sands (S6 to S8) are compared, beach sands contain high amount of the studied elements (Fig. 3).

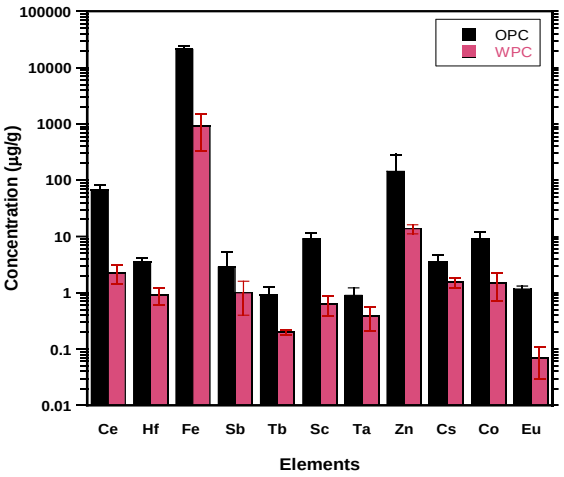

Fig. 2 *Comparison of average concentrations of the studied elements in ordinary Portland cements (OPC) and white Portland cements (WPC).*

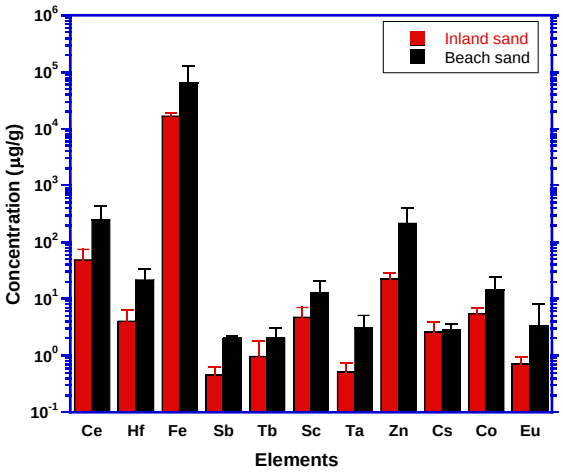

Fig. 3 *Comparison of average concentrations of the studied elements in inland sands and beach* sands.

The average concentration of Garnet, Magnetite plus Illmenite, Zircon, Rutile and Monazite in Cox'sBazar beach sands are 46.9%, 38.1%, 6.40%,1.19% and 0.95%, respectively [26]. These heavy minerals are mainly responsible for high concentrations of the studied elements in beach *sands.* For example, Zircon and monazite minerals contain high amounts of rare earth elements and, Magnetite and Illmenite minerals are the main source of Fe in the beach sands.

Table 1. *Nuclear data for long-lived radionuclide induced in nuclear radiation shielding (JENDL 3.3) [24].*

Activation product[1]	**Nuclear reaction**	**Target abundance (%)**	**Half-life**	**Gamma-ray Energy (keV)**	**Gamma-ray Intensity (%)**
^{141}Ce	^{140}Ce(n,γ)^{141}Ce	88.45	32.5 d	145	48
^{181}Hf	^{180}Hf(n,γ)^{181}Hf	35.08	42.4 d	482	81
^{59}Fe	^{58}Fe(n,γ)^{59}Fe	0.282	44.5 d	1099	56.5
^{124}Sb	^{123}Sb(n,γ)^{124}Sb	42.79	60.2d	1691	47.57
^{160}Tb	^{159}Tb(n,γ)^{160}Tb	100	72.3 d	299	26.13
^{46}Sc	^{45}Sc(n,γ)^{46}Sc	100	83.8 d	889	99.98
^{182}Ta	^{181}Ta(n,γ)^{182}Ta	99.99	114.7 d	1221	27.23
^{65}Zn	^{64}Zn(n,γ)^{65}Zn	48.63	243.9 d	1116	50.04
^{134}Cs	^{133}Cs(n,γ)^{134}Cs	100	2.07 y	796	85.46
^{60}Co	^{59}Co(n,γ)^{60}Co	100	5.27 y	1173 1332	100 100
^{154}Eu	^{153}Eu(n,γ)^{154}Eu	52.3	8.5 y	123 1275	40 35
^{152}Eu	^{151}Eu(n,γ)^{152}Eu	47.81	13.5 y	121 1408	39.8 29.2

[1]Radionuclides are arranged according to their increasing half-lives.

Table 2. *Concentrations of the determined elements in cements in µg/g, except as indicated (%).*

Element	Elemental concentration						
	OPC1	OPC2	OPC3	OPC4	OPC5	OPC6	Mean±SD[1]
Ce	63.6±1.7	75.0±2.0	72.3±1.9	70.9±1.9	83.1±2.1	41.9±1.3	67.8±14.2
Hf	2.83±0.14	4.05±0.18	3.59±0.17	4.08±0.19	3.95±0.18	2.59±0.13	3.51±0.65
Fe (%)	1.96±0.04	2.45±0.05	2.10±0.05	2.07±0.05	2.41±0.05	1.68±0.04	2.11±0.29
Sb	0.587±0.107	4.77±0.54	1.25±0.17	7.10±0.77	1.66±0.23	1.66±0.22	2.84±2.54
Tb	1.58±0.08	0.833±0.048	0.859±0.049	0.739±0.044	0.813±0.047	0.563±0.035	0.898±0.350
Sc	9.13±0.09	11.3±0.1	5.13±0.06	10.04±0.11	11.5±0.1	7.03±0.08	9.02±2.51
Ta	0.764±0.083	1.23±0.13	0.957±0.103	0.763±0.087	1.26±0.13	0.412±0.054	0.897±0.321
Zn	57.7±4.3	286±16	39.8±3.6	364±19	63.2±4.7	57.0±4.3	144±142
Cs	2.92±0.13	5.12±0.19	3.39±0.14	3.02±0.14	4.41±0.17	2.15±0.10	3.50±1.08
Co	7.18±0.33	10.2±0.4	7.88±0.37	13.4±0.6	10.7±0.5	5.12±0.26	9.08±2.93
Eu	1.19±0.04	1.35±0.04	1.21±0.04	1.14±0.04	1.22±0.04	0.883±0.029	1.16±0.15

[1]SD means standard deviation (1σ). Uncertainties with individual data are due to counting statistics (1σ) whereas with mean values are standard deviation (1σ).

Table 3. *Concentrations of the determined elements in white Portland cements and heavy minerals in µg/g, except as indicated (%).*

Element	Elemental concentration						
	WPC1	WPC2	WPC3	Mean±SD	Zircon	Magnetite	Illmenite
Ce	1.75±0.75	1.83±0.21	3.24±0.42	2.27±0.84	467±9	40.7±1.1	67.2±1.5
Hf	1.23±0.09	0.613±0.101	0.885±0.115	0.910±0.310	12534±349	8.06±0.28	12.1±0.4
Fe (%)	0.135±0.012	0.024±0.007	0.117±0.019	0.090±0.058	0.097±0.007	74.5±1.1	47.5±1.0
Sb	1.65±0.03	0.456±0.041	0.892±0.094	1.00±0.60	0.650±0.131	0.457±0.104	1.68±0.24
Tb	0.213±0.019	0.218±0.023	0.176±0.024	0.202±0.023	24.9±0.7	0.937±0.035	1.60±0.05
Sc	0.484±0.103	0.495±0.030	0.921±0.051	0.633±0.249	69.5±0.5	5.03±0.06	36.6±0.3
Ta	0.201±0.037	0.533±0.101	0.394±0.087	0.376±0.167	8.79±0.70	1.13±0.11	40.5±3.1
Zn	16.5±2.1	117±20	12.9±5.2	13.7±2.5	51.3±4.8	407±24	267±16
Cs	1.27±0.12	1.48±0.17	1.87±0.23	1.54±0.30	1.40±0.09	0.588±0.054	1.32±0.08
Co	1.49±0.10	0.729±0.120	2.23±0.25	1.48±0.75	1.39±0.13	50.7±1.8	40.4±1.5
Eu	0.024±0.001	0.081±0.011	0.105±0.015	0.070±0.042	2.43±0.19	0.662±0.068	0.904±0.082

Uncertainties with individual data are due to counting statistics (1σ) whereas with mean values are due to standard deviation (1σ).

Table 4. *Concentrations of the studied elements in sands in μg/g, except as indicated (%).*

Element	Elemental concentration									
	S1	S2	S3	S4	S5	Mean±SD	S6	S7	S8	Mean±SD
Ce	11.5±0.6	53.0±1.5	41.4±1.3	84.4±2.2	53.5±1.6	48.8±26.2	80.7±1.80	215±14	456±21	251±190
Hf	2.33±0.13	3.87±0.18	2.56±0.13	8.23±0.32	3.22±0.16	4.04±2.42	8.98±0.31	22.4±2.6	33.5±3.7	21.6±12.3
Fe (%)	1.67±0.04	1.89±0.04	1.18±0.03	1.80±0.04	1.76±0.04	1.66±0.28	2.15±0.04	3.59±0.05	13.5±1.6	6.41±6.18
Sb	0.468±0.099	0.740±0.129	0.271±0.071	0.359±0.084	0.405±0.093	0.449±0.178	2.09±0.29	1.78±0.12	2.22±0.13	2.03±0.23
Tb	0.160±0.016	0.523±0.033	0.488±0.032	2.09±0.10	1.57±0.08	0.967±0.823	1.03±0.04	1.88±0.12	3.07±0.15	1.99±1.03
Sc	1.28±0.03	7.07±0.08	4.55±0.06	3.87±0.05	6.76±0.08	4.71±2.36	5.68±0.06	12.5±0.5	20.6±0.7	12.9±7.47
Ta	0.329±0.049	0.542±0.067	0.361±0.051	0.394±0.054	0.921±0.101	0.509±0.244	1.21±0.12	2.90±0.13	5.24±0.12	3.12±2.03
Zn	23.6±2.6	23.5±2.5	12.4±1.7	26.5±2.7	26.8±2.8	22.5±5.9	300±18	3.05±0.72	341±15	215±184
Cs	0.848±0.061	4.28±0.17	2.67±0.12	1.44±0.08	3.51±0.15	2.55±1.42	2.20±0.11	1.65±0.12	2.67±0.12	2.84±0.76
Co	6.18±0.30	6.94±0.32	2.92±0.18	5.66±0.28	5.76±0.29	5.49±1.52	8.43±0.40	8.05±0.30	26.5±0.7	14.3±10.5
Eu	0.332±0.040	0.818±0.071	0.628±0.059	0.930±0.079	0.826±0.074	0.707±0.236	1.04±0.09	0.134±0.021	8.92±0.16	3.36±4.83

Uncertainties associated with individual values are due to counting statistics (1σ) whereas with mean values are standard deviation (1σ).

Altough heavy minerals contain high amounts of the elements responsible for long-lived induced radioactivity, they are sometimes used to construct nuclear shielding to increase density of the shielding concrete up to the required level [14]. Elemental concentrations of the studied elements in the most abundant heavy minerals of the Cox's Bazar beach sands– Zircon, Magnetite and Illmenite are given in Table 4. Among the three studied heavy minerals, zircon contains high contents of REEs (Sc, Ce, Eu, Tb) which will potentially contribute to the long-lived radionuclides in the concrete (Table 4). Illmenite having high abundances of Co and Zn contribute to the heavy concrete shield when they are. used as ingredients for reactor shielding materials. Since ^{152}Eu, ^{154}Eu and ^{60}Co contribute 99-100% of the total residual radioactivity in terms of clearance level (CL) value induced in ordinary concrete at the time of decommissioning [27, 6-7], Zircon and Illmenite have the potential to increase long-lived radionuclides when used as ingredients in nuclear shielding materials. The valuable heavy sand mineral separation into individuals is accomplished based on the specific gravity, particle size, electrical conductivity and magnetic susceptibility of the heavy minerals. All the mineral products may contain traces of elements of interest due to incomplete separation of monazite. Monazite mineral contains REE and actinides. Therefore, extra care should be taken to choose heavy minerals for using as biological shielding ingredients of a nuclear installation to reduce induced radioactivity.

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4. Conclusion

Since long-lived radionuclides formed by neutron-induced reactions in inner part of the biological concrete shielding cause problem in waste management of the activated shielding, it is necessary to choose ingredients for reactor shielding with minimum concentration of the elements which are responsible of long-lived radionuclides. In this study, elemental concentrations of such elements Ce, Hf, Fe, Sb, Tb, Sc, Ta, Zn, Cs, Co and Eu in cements, sands and heavy minerals separated from beach sands are reported so that a strategy can be made to develop low-activation concrete shielding using local shielding materials. Average concentrations of the studied elements in WPC are much lower in comparison with OPC. This study reveals that Inland sands contain low concentrations of the studied elements than those of beach sands. Beach sands contain heavy minerals which are mainly responsible for high concentration of the elements causing high residual activity. Elemental compositional data of the studied local shielding materials can be used to choose ingredients necessary for constructing radiation shielding especially for the future NPP projects in Bangladesh.

The valuable heavy sand mineral separation into individuals is accomplished based on the specific gravity, particle size, electrical conductivity and magnetic susceptibility of the heavy minerals.

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Conflicts of Interest

The authors have no conflict of interest.

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