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Determination of ^{234}U, ^{235}U, ^{238}U, ^{228}Th, ^{230}Th, ^{232}Th, ^{226}Ra, ^{228}Ra, and ^{210}Pb in foods from Brazilian Total Diet

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Abstract This study determined the concentrations of ^{234}U, ^{235}U, ^{238}U, ^{228}Th, ^{230}Th, ^{232}Th, ^{226}Ra, ^{228}Ra, and ^{210}Pb in 82 types of food, grouped into 19 groups according to the Brazilian Total Diet, which reflects the dietary habits of the São Paulo State population. Radionuclides were quantified using Ultra low level alpha and beta total counting, and alpha spectrometry, after radiochemical separations. The results of this preliminary work showed that the food groups analyzed gave a consumption of effective dose lower than the dose limit for the public, of 1 mSv year^{-1}.

Keywords Total Diet · Natural radionuclides · Ultra low level alpha and beta total counting · Alpha spectrometry

Introduction

The World Health Organization (WHO) recommends the Total Diet study (TDS) as being the most suitable method to estimate the dietary intakes of contaminants and nutrients for a country or a large population area [1].

Several studies have evaluated the presence of nutrients and contaminants in foods, in order to provide healthy diet for the population [1–5].

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Many chemical substances consumed have importance, not only for their nutritional perspective, but also by the harmful effects causing serious health problems such as cancer, birth defects and brain damage [6].

The National Household Food Budget Survey (POF) of the Brazilian Institute of Geography and Statistics (IBGE) is used to gather data on food consumption for a population [5, 7, 8].

The southeastern region of Brazil has the highest demographic concentration. Food supply to this region comes from all parts of the country. Therefore, it is of outstanding importance to determine the levels of natural radionuclides from the uranium and thorium series in food from this region [9].

The presence of radioactivity has been detected in food and water in several parts of the world. The natural radionuclide concentrations vary according to several factors, such as local geology, climate and agricultural practices [10]. The degree of damage to human health depends on the type of radionuclide and the period of time that people are exposed to it [11].

It is known that food and water contain radioactive elements, which contribute to an effective internal dose after ingestion [11, 12]. Natural radionuclides from the uranium and thorium series occur widely distributed in the earth's crust [13].

The radioactivity measurement in foods and the environment is extremely important to monitor the radiation levels to which man can be directly or indirectly exposed. Some foods have the ability to retain radionuclides, such as natural radioisotopes and other contaminants [14, 15].

The determination of low concentrations of these elements in food samples is time consuming and requires tedious chemical procedures. An essential feature of these methods is the pre-concentration and purification of the

radionuclides of interest. This is important to isolate them from the large amounts of inactive substances present in the sample and also to separate them from radioisotopes that may interfere in these determinations [16].

In this study, the TDS was based on the evaluation of food representing a food list, which reflects the dietary habits of the Southeastern region population. The food consumption data and information were obtained from the National Household Food Budget Survey conducted by the Brazilian Institute of Geography and Statistics in 2008–2009. The food list was based on the personal consumption over a 24-hour period, of food consumed both in and out of the household. The list of foods consumed used in this study, was undertaken in the southeastern region of Brazil: 14,078 households being surveyed, 3064 were randomly selected as the sub-sample for the Analysis of Personal Food Consumption to obtain data of 7302 inhabitants age ten or more, in the urban and rural areas of the region. The food consumption in and out of the households in two non-consecutive days was registered [17]. The present food list was then composed by 82 foods divided in 19 food groups.

Experimental

Food group preparation

(1) Only 11 food groups were analyzed in this paper: cattle meat, pork, poultry, industrialized meat and offal, fruits, vegetables, beans, cereals, dairy products, eggs, cakes and biscuits.
(2) The food groups were prepared table-ready, which means that if necessary the food was prepared following the normal preparation habits of the studied population. The preparation includes discarding non edible portions (bones, fat, fruit seeds, etc.), cooking when necessary. No seasonings, oils or other condiments were added in the preparation.
(3) After the table-ready preparation, similar samples were mixed to form the groups. They were then homogenized, freeze-dried and grounded to a fine powder.

Experimental procedures

Approximately 10 g of food sample were burnt in order to obtain ash at 450 °C for 24 h in a muffle furnace. After that, the ash samples were dissolved with three concentrate acids (nitric, perchloric, and hydrofluoric). The final solution was evaporated and the salts dissolved with 8 M nitric acid. The solution was divided into two parts in order to determine all radioisotopes.

Ra and Pb determination

(1) These radioisotopes were purified by radiochemical separation, with Pb^{2+} (20 mg mL^{-1}) and Ba^{2+} (20 mg mL^{-1}) used as carriers to determine loss during the analysis. The radionuclide was coprecipitated as $Ba(Ra)SO_4$ and $PbSO_4$ using 3 M sulfuric acid, solution of 1 M citric acid with 0.1 % phenol and concentrated ammonium hydroxide at pH 6.5 using methyl orange as indicator.
(2) After that, the precipitate was dissolved with 2 g of EDTA and 5 mL of concentrated ammonium hydroxide. The ^{226}Ra and ^{228}Ra were selectively coprecipitated with addition of 5 mL of 25 mg/mL ammonium sulphate and concentrated glacial acetic acid. The supernatant was saved to ^{210}Pb determination.
(3) The Ra precipitate was filtered through a 0.45 μm Millipore membrane. The samples were stored for 30 days to await the secular equilibrium between ^{226}Ra and ^{222}Rn, and ^{228}Ra and ^{228}Ac.
(4) The ^{210}Pb containing in the supernatant was precipitated using 1 mL of 1 M Na_2S. The precipitate was dissolved with 5 M HNO_3, evaporated to dryness and the salts dissolved with deionized water. The Lead was finally precipitated in the form of $PbCrO_4$ with addition of 40 % ammonium acetate and solution of 30 % Na_2CrO_4.
(5) The precipitate formed was filtered through a 0.45 μm Millipore membrane. The samples were stored for 30 days to wait the secular equilibrium between ^{210}Pb and ^{210}Bi.
(6) The chemical recovery for Ra and Pb were obtained by gravimetry.
(7) The ^{226}Ra, ^{228}Ra and ^{210}Pb were quantified using an ultra low level alpha and beta total counting with a gaseous flow proportional detector model S5-XLB Tennelec from Canberra Industries during 120 min.

U and Th determination

(1) A tracer was added in the other fraction of the sample solution containing uranium and thorium ^{229}Th and ^{232}U in order to determine the chemical recuperation.
(2) Radiochemical Th separation was carried out using Dowex 1 × 2 anionic exchange resin pre-conditioned with 8 M nitric acid. The sample in nitric media was percolated through anionic column Dowex 1 × 2 resin. Thorium was retained and the uranium followed to the effluent. Thorium was eluted with concentrated hydrochloric acid. The eluted solution was dried on a

hot plate and the salts dissolved with the electroplating solution at pH 2.3. The electroplating solution was prepared using concentrated sulfuric acid, 0.3 M sodium sulfate and deionized water.

(3) The effluent containing U was dried, the salts dissolved with 3 M HNO_3, and the solution was percolated through a chromatography column UTEVA pre-conditioned with 3 M HNO_3. The effluent was discarded. After that 9 M HCl was added through the column to modify the medium, the effluent was discarded and U was then eluted with 0.01 M HCl. The effluent was dried on a hot plate and the salts dissolved with 3 M sulfuric acid and 0.8 M ammonium sulfate.

(4) The radioisotopes were electrodeposited on polished silver plates using an electrical current of 1.0 A to thorium and 1.2 A to uranium during 60 mins.

(5) The U and Th isotopes were analyzed in an Alpha Analyst spectrometer with 12 passivated implanted planar silicon (PIPS) detectors (counting efficiency 18 %), and Genie 2000/Alpha Analyst spectroscopy systems, from Canberra Industries during 200,000 s.

(6) Alpha particles energies 5.41 MeV for the ^{228}Th, 4.90 MeV for the ^{229}Th tracer, 4.67 MeV for the ^{230}Th, and 4.01 MeV for ^{232}Th were used to quantify thorium.

(7) Alpha particles energies 4.31 MeV for the ^{232}U tracer, 4.74 MeV for the ^{234}U, 4.47 MeV for the ^{235}U, and 4.19 MeV for ^{238}U were used to quantify uranium.

Results and discussion

The Reference Material IAEA-Soil 327 [18] was analyzed in order to verify the quality control of the results, knowing that the soil is much more matrix complex than foods. The activity concentrations of ^{226}Ra, ^{228}Ra, ^{210}PB, Th and U radioisotopes in the IAEA-Soil 327 obtained after radiochemical separations are presented in the Table 1.

The results obtained for the analysis of reference material IAEA-Soil 327 showed that the methods applied to determine natural radioisotopes after radiochemical separation and counting of total alpha and beta emitters were reliable and accurate.

This study presents preliminary results, as the analyzed groups represent only 50.4 % of the food groups of the 2nd Brazilian Total Diet study.

The chemical recovery of Ra isotopes were between 77 and 90 % and of Pb was between 50 and 65 %.

The chemical recovery of thorium analysis varied from 85 to 100 %. The results obtained for ^{230}Th and ^{232}Th activities for freeze-dried food samples were below the detection limit, being below 0.17 Bq kg^{-1} for ^{230}Th, and below 0.12 Bq kg^{-1} for ^{232}Th.

The chemical recovery of the uranium analysis varied from 60 to 100 %. The results obtained for uranium isotopes activity for all freeze-dried food samples were below the detection limit, being below 0.14 Bq kg^{-1} for ^{234}U and ^{238}U, and below 0.26 Bq kg^{-1} for ^{235}U.

Table 2 presents the results obtained for determinations of ^{226}Ra, ^{228}Ra, ^{210}Pb and ^{228}Th activities in the freeze dried food groups.

Table 3 presents the results obtained for the determinations of ^{226}Ra, ^{228}Ra, ^{210}Pb and ^{228}Th activities in the table-ready food groups which took into consideration the water loss during the freeze-dried process when needed.

Table 4 presents daily intakes of ^{226}Ra, ^{228}Ra, ^{210}Pb and ^{228}Th in food groups. For these results the daily consumption per capita for each food group was considered. The values less than the detection limit not entered in the calculation of total activity for ^{228}Ra and ^{210}Pb.

The doses from intakes of radionuclides in foods by population groups can be calculated from measurements of concentrations of radionuclides in foods, consumption rates and dose coefficients. These can be described by the following Eq. (1) [19]:

$$E_A = \sum_j \sum_f C_{fj} \times M_{jA} \times T \times h_{jA} \quad (1)$$

where E_A is the committed effective dose (mSv) per group. C_{fj} is the average concentration (Bq kg^{-1}) of radionuclide j on or within food. M_{jA} is the mass (kg day^{-1}) of food item f consumed per day by the A group. T is the total time (days) considered. h_{jA} is the ingestion dose coefficient (mSv Bq^{-1}) of radionuclide j for the A group.

Table 5 presents the radionuclide concentration contributors to the annual effective dose by ingestion the ^{226}Ra, ^{228}Ra, ^{210}Pb, and ^{228}Th, considering the calculation of the mass of food 0.768 kg of the daily ingestion per adult.

The radiochemical separation followed by alpha and beta total counting, in a gaseous flow proportional counter of ultra-low background or followed by alpha spectrometry was demonstrated to be a sensitive and accurate method for determination of natural isotopes of the uranium and thorium series.

In general, the decreasing order of radionuclide concentrations observed in this study is in agreement with what is generally found in the literature on Brazilian food:^{226}Ra > ^{210}Pb > ^{228}Ra > ^{232}Th ≥ ^{238}U [20].

It is known that thorium is an element of low solubility, but the results obtained in this work presented values of ^{228}Th higher than ^{232}Th. This imbalance can be explained by the fact that ^{228}Th originates from ^{228}Ra, which has higher solubility [21].

Table 1 Results of ^{226}Ra, ^{228}Ra, ^{210}Pb and Th radioisotopes in IAEA-Soil-327

Radionuclide	Activity ± uncertainty (Bq kg^{-1})	Certified value (Bq kg^{-1})	95 % CI	Relative error (%)
^{226}Ra	36.5 ± 1.3	34.1	32.7–35.5	7.0
^{228}Ra	35.0 ± 4.0	38.7	37.8–39.6	9.6
^{210}Pb	49.0 ± 7.0	58.8	53.9–63.7	17
^{234}U	33.4 ± 1.7	31.9	30.4–33.4	4.7
^{235}U*	1.6 ± 0.2	1.4	1.22–1.64	14
^{238}U	33.2 ± 1.7	32.9	31.4–34.2	0.9
^{228}Th	37.0 ± 1.7	38.2	37.2–39.2	3.1
^{230}Th	33.3 ± 1.5	34.1	32.4–35.8	2.3
^{232}Th	37.6 ± 1.7	38.7	37.2–40.2	2.8

* Information value

Table 2 Results of ^{226}Ra, ^{228}Ra, ^{210}Pb and ^{228}Th determinations in the freeze-dried food groups

Sample (groups)	^{226}Ra (Bq kg^{-1})	^{228}Ra (Bq kg^{-1})	^{210}Pb (Bq kg^{-1})	^{228}Th (Bq kg^{-1})
Cakes and biscuits	6.6 ± 1.5	3.4 ± 0.6	<4.0	1.0 ± 0.1
Industrialized meat and offal	5.2 ± 1.4	4.9 ± 0.5	<4.0	0.8 ± 0.1
Fruits	5.9 ± 1.4	<2.0	<4.0	0.8 ± 0.1
Beans	4.5 ± 1.2	4.0 ± 0.3	4.2 ± 0.4	1.0 ± 0.1
Vegetables	8.1 ± 1.6	5.9 ± 1.9	<4.0	2.2 ± 0.1
Eggs	7.0 ± 1.6	5.2 ± 1.2	13.5 ± 1.7	1.2 ± 0.1
Cattle meat	5.0 ± 1.4	<2.0	9.7 ± 1.6	1.2 ± 0.1
Cereals	4.2 ± 1.3	2.0 ± 0.4	9.2 ± 1.5	1.0 ± 0.1
Poultry	4.5 ± 1.3	2.1 ± 0.6	<4.0	0.9 ± 0.1
Dairy products	3.3 ± 1.2	7.1 ± 0.3	<4.0	1.1 ± 0.1
Sweets	4.3 ± 1.3	10.1 ± 1.2	<4.0	12 ± 0.1

Table 3 Results of ^{226}Ra, ^{228}Ra, ^{210}Pb and ^{228}Th determinations in the table-ready food groups

Sample (groups)	^{226}Ra (Bq kg^{-1})	^{228}Ra (Bq kg^{-1})	^{210}Pb (Bq kg^{-1})	^{228}Th (Bq kg^{-1})
Cakes and biscuits	6.6 ± 1.5	3.4 ± 0.6	<4.0	1.00 ± 0.10
Industrialized meat and offal	2.5 ± 0.7	2.3 ± 0.2	<1.9	0.38 ± 0.05
Fruits	1.3 ± 0.3	<0.5	<0.9	0.18 ± 0.02
Beans	1.0 ± 0.3	0.9 ± 0.1	1.0 ± 0.1	0.23 ± 0.02
Vegetables	1.0 ± 0.2	0.7 ± 0.2	<0.5	0.27 ± 0.01
Eggs	1.7 ± 0.4	1.3 ± 0.3	3.3 ± 0.4	0.29 ± 0.02
Cattle meat	2.3 ± 0.6	<0.9	4.5 ± 0.7	0.56 ± 0.05
Cereals	1.2 ± 0.4	0.6 ± 0.1	2.7 ± 0.4	0.30 ± 0.03
Poultry	1.5 ± 0.2	0.7 ± 0.2	<1.4	0.31 ± 0.03
Dairy products	0.5 ± 0.2	1.16 ± 0.05	<0.7	0.18 ± 0.02
Sweets	4.3 ± 1.3	10.1 ± 1.2	<4	12.0 ± 0.1

Background levels of radionuclides in foods are dependent on several factors, including the type of food and the geographic region where they have been produced. There are other natural radioisotopes that exist in much lower concentrations, and originate from the decay of uranium and thorium. Once in the environment, radioactive material can also become incorporated into food as it is taken up by plants, or ingested by animals [11].

The risk associated with an intake of radionuclides in the body is proportional to the total dose delivered by the radionuclides. In general it is assumed that stochastic effects occur linearly with dose and usually the effective

Table 4 Daily intakes of ^{226}Ra, ^{228}Ra, ^{210}Pb and ^{228}Th

Sample (food groups)	Daily consumption per capita (kg)	^{226}Ra (Bq kg^{-1})	^{228}Ra (Bq kg^{-1})	^{210}Pb (Bq kg^{-1})	^{228}Th (Bq kg^{-1})
Cakes and biscuits	0.0299	1.97×10^{-1}	1.02×10^{-1}	$<1.20 \times 10^{-1}$	3.00×10^{-2}
Industrialized meat and offal	0.0143	3.50×10^{-2}	3.30×10^{-2}	$<2.70 \times 10^{-2}$	5.00×10^{-3}
Fruits	0.0819	1.10×10^{-1}	$<3.70 \times 10^{-2}$	$<7.50 \times 10^{-2}$	1.50×10^{-2}
Beans	0.224	2.29×10^{-1}	2.03×10^{-1}	2.13×10^{-1}	5.10×10^{-2}
Vegetables	0.0785	7.80×10^{-2}	5.70×10^{-2}	$<3.90 \times 10^{-2}$	2.10×10^{-2}
Eggs	0.0099	1.70×10^{-2}	1.30×10^{-2}	3.20×10^{-2}	3.00×10^{-3}
Cattle meat	0.0067	1.60×10^{-2}	$<6.00 \times 10^{-3}$	3.00×10^{-2}	4.00×10^{-3}
Cereals	0.191	2.37×10^{-1}	1.13×10^{-1}	5.20×10^{-1}	5.60×10^{-2}
Poultry	0.033	5.10×10^{-2}	2.40×10^{-2}	$<4.50 \times 10^{-2}$	1.00×10^{-2}
Dairy products	0.076	4.10×10^{-2}	8.80×10^{-2}	$<5.00 \times 10^{-2}$	1.40×10^{-2}
Sweets	0.0238	1.02×10^{-1}	2.40×10^{-1}	9.50×10^{-2}	2.86×10^{-1}
Total	0.768	1.113	0.873*	0.890*	0.495

* The values less than the detection limit not entered in the calculation of total activity

Table 5 Radionuclide concentration contributors to the annual effective dose

Radionuclide	Concentration (Bq kg^{-1})	Ingestion dose coefficient* (mSv Bq^{-1})	Dose (mSv year^{-1})
^{226}Ra	1.113	2.8×10^{-4}	8.74×10^{-2}
^{228}Ra	0.873	6.9×10^{-4}	1.69×10^{-1}
^{210}Pb	0.890	6.9×10^{-4}	1.72×10^{-1}
^{228Th}	0.495	7.2×10^{-5}	1.00×10^{-2}
Effective dose total	4.39×10^{-1} mSv year^{-1}		

* Committed effective doses for ingestion for adults [19]

dose is used to define this risk. Therefore, it is a parameter for the biological effect [22]. The committed effective doses received by members of the public who consume the food groups analyzed in this work are lower than the dose limit for the public, of 1 mSv year^{-1}.

The measurement of radioactivity in the food groups of the Total Diet is extremely important to monitor levels of radiation to which man can be directly or indirectly exposed, in particular, the determination of the natural radionuclides of the uranium and thorium series.

Conclusions

The study was able to determine radionuclides from the natural series, ^{234}U, ^{235}U, ^{238}U, ^{228}Th, ^{230}Th, ^{232}Th, ^{226}Ra, ^{228}Ra, and ^{210}Pb in food groups of the 2nd Brazilian Total Diet study. The results showed low radioactivity concentrations and also low committed effective dose by intake, below the dose limit for the public, of 1 mSv year^{-1}.

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