Characterization of Human Exposure to Mineral Sands Dust in a Brazilian Village

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Abstract The aim of this study was to characterize human exposure to mineral dust particles using PIXE (Particle Induced X rays Emission) and ^{252}Cf-PDMS (Plasma Desorption Mass Spectrometry) techniques. The dust particles were generated during the separation process of mineral sands to obtain rutile, ilmenite, zircon and monazite concentrates. The aerosol samples were collected at the village and during the process to concentrate ilemenite. A cascade impactor with six stages was used to collect mineral dust particles with aerodynamic diameter in the range of 0.64 to 19.4 μm. The particles impacted on each stage of the cascade impactor were analyzed by PIXE (Particle Induced X ray Emission) and the elemental mass concentration and the MMAD (Mass Median Aerodynamic Diameter) were determined. Employing the ^{252}Cf-PDMS technique the chemical compound present in aerosols particles and in urine samples were identified. The mass spectra (^{252}Cf-PDMS technique) of dust samples showed the presence of the thorium silicate, thorite and zircon in the fine fraction of aerosol. The ^{252}Cf-PDMS technique was, also, used to characterize urine sample from a inhabitant of the village. The results show that Buena village inhabitants inhale mineral sands dust particles. Based on the results from the lichen samples it could be concluded that at least during the last 15 years the inhabitants of the village have been exposed to monazite particles. Results suggest that the there is natural source of aerosol particles containing ^{226}Ra and ^{210}Pb (probably the swamp) besides the mineral sands dust.

KEYWORDS: Minerals sands, human exposure, Thorium, ^{252}Cf-PDMS, PIXE

1. Introduction

People living in regions with high concentration of monazite can incorporate metals, specially thorium. The incorporation can be by ingestion (local foodstuff and coarse particles) and by inhalation of fine airborne particles. Workers involved in mineral concentration processes inhale mineral particles generated during the mineral processing [1, 2].

The most important Brazilian mineral sands deposit and processing plant are located in Buena, a seashore village with 300 inhabitants in the North of Rio de Janeiro states.

^{232}Th average concentration in feces and urine samples from a group of Buena village inhabitant were 6.7±1.6 mBq/gash and 1.5±0.5 mBq/L respectively [3]. ^{232}Th concentrations in two daily complete meals consumed by inhabitants were respectively 42.0 and 15.0 mBq/kgwet [4]. The thorium concentrations in the meals do not explain the concentration of ^{232}Th observed in inhabitant's feces samples.

In order to evaluate the risk to the human beings due to dust particles inhalation, it is necessary to determine the deposition rate, the concentration and the kinetics of the particles flowing in the respiratory

tract. Furthermore, the chemical composition, size and the elemental mass concentration of the particle in the respirable fraction of aerosol are necessary for evaluation of the worker's risk [5, 6, 7, 8].

The airborne-particle samples in fine fraction of aerosols (aerodynamic diameters (daer) < 2.4 μm [9]) were collected at the residences using a cascade impactor, a stack filter unit and a portable air sampler with cyclone. The elemental mass concentration and the Mass Median Aerodynamic Diameter (MMAD) of the aerosol particles were determined by PIXE (Particle Induced X rays Emission) technique. Concentrations of ^{232}Th, ^{228}Th, ^{228}Ra, ^{238}U, ^{234}U, ^{226}Ra and ^{210}Pb in aerosol and in lichens samples were determined by the alpha spectrometry technique.

2. Experimental method

Aerosol samples were collected at the village and at the mineral sands processing plant during the reprocessing of ilmenite to obtain concentrates of ilmenite. The airborne particle samples were collected using a six-stage cascade impactor (CI) and a stack filter unit [10]. The air samplers were placed at 1.5 m from the ground, running 8 hours per day during 10 days (from 8:30 up to 16:30 h) the dry season. Airborne particles samples were analyzed by PIXE using 2 MeV proton beam obtained from the 4 MV van de Graaff accelerator at PUC-RIO. The X-Rays were detected by a Si-PIN detector with two geometries: one using 0.2 mm - thick aluminum foil as an X-Rays absorber and the second one without aluminum foil. The efficiency curve was determined using reference standard material. The X-Rays spectra were analyzed using an updated version of custom-designed software based on the stripping of a multi-elemental spectrum [10,11].

Lichens (Ramalina gracílis (Pres.) Nyl.,) samples were used as an indicator of the inhabitants exposure to thorium and uranium bearing particles during the last 15 years. These lichens were 15 years old and were collected in two villages: Buena and Manguinhos. The Manguinhos village, located approximately 5 km far way from the Buena village, was used as a control area for lichen samples.

^{232}Th, ^{228}Th, ^{228}Ra, ^{238}U, ^{234}U, ^{226}Ra and ^{210}Pb concentrations were determined in the aerosol samples collected by AGF and cyclone samplers, and in the lichen samples. These samples were analyzed in the Laboratory for Aerosol Characterization of Instituto de Radioproteção e Dosimetria – IRD/CNEN. A sequential analytical method for the determination of these radionuclides was used. Uranium and thorium isotopes were initially separated using tri-n-octyl phosphine oxide (TOPO) supported on a column of silica gel. The uranium isotopes were extracted with TOPO onto a polymeric membrane. Thorium isotopes were co-precipitated with lanthanum fluoride. ^{232}Th, ^{228}Th, ^{228}Ra, ^{238}U and ^{234}U concentrations were determined by alpha spectrometry [12]. The alpha spectra were obtained by an EG&G ORTEC model 576 system.

Radium isotopes and lead dissolved in the solution were separated by co-precipitation with mixed barium and lead sulphate and centrifugation. The ^{228}Ra, ^{226}Ra and ^{210}Pb concentrations were determined with a Bertold model LB770 alpha and beta counter system [13].

Urine samples of an inhabitant living in Buena village for more than 30 years, but not involved in the mineral concentration processes, were collected. This sample (1.4 L) was collected during 24 hours, homogenized and an aliquot of 750 mL was obtained and divided up to an aliquot of 35 mL, a volume of 10 μL was deposited on a stainless steel cube and dried at room temperature to produce a urine film. The urine film was analyzed using the ^{252}Cf-PDMS system of the Chemical Characterization Laboratory of the Texas A&M University (ATM) [14]. The aerosol collected at 6th stage of the cascade impactor, the concentrate of monazite and the cerium oxide were analyzed by ^{252}Cf-PDMS at PUC-Rio [15,16].

3. Results and discussion

The elemental mass concentrations and MMADs of the airborne dust particle samples collected at the plant and at the village (residences number 1, 2 and 3) are shown in Tables 1 and 2, respectively. The uncertainly associated to the mass concentration is about 10 % (volume and mass determination). The geometric standard deviation is lower than 3 for all MMAD values. At the plant, the highest Th concentration occurs during the electromagnetic separation process however the cerium concentration was below the detection limit of PIXE method (4.3 ng). These results suggest that monazite dust particles were not present in the respirable fraction of aerosol and that the thorium bearing particles were due to other aerosol source. The presence of Ti and Fe in the dust particle X ray spectra suggesting that rutile and ilmenite were the source of Fe and Ti bearing particles. At the village, the MMAD values were less than 2.5 μm and the elemental mass concentrations decreases depending on the relative plant to house distance and wind direction. This suggests the mineral sands plant the main source of airborne particles in the respirable fraction of the aerosol.

Table 1. Elemental mass concentration in the respirable fraction of aerosol ($\mu g/m^3$).

	Elements																		
Local	**Cl**	**K**	**Ca**	**Ti**	**V**	**Cr**	**Mn**	**Fe**	**Co**	**Ni**	**Cu**	**Zn**	**Br**	**Zr**	**Ce**	**La**	**Pb**	**Th**	**Y**
A	<dl	<dl	0.89	0.52	<dl	0.25	0.10	0.53	<dl	0.45	<dl	0.01	<dl	0.04	<dl	<dl	0.01	0.02	<dl
B	<dl	<dl	3.58	.03	<dl	2.42	0.11	3.81	<dl	0.03	2.36	0.46	<dl	8.23	<dl	<dl	0.09	0.14	<dl
	<dl	<dl	3.75	5.77	<dl	1.59	0.14	5.25	<dl	0.03	<dl	0.03	<dl	0.36	<dl	<dl	0.05	<dl	<dl
C	<dl	<dl	3.41	2.33	<dl	2.63	2.67	3.18	<dl	0.03	0.02	0.02	<dl	<dl	<dl	<dl	<dl	<dl	<dl
R1	1.6E5	710	270	1.78	0.08	0.03	0.05	4.56	0.03	0.01	0.01	0.05	5E-3	0.14	0.08	0.06	0.06	0.03	1E-2
R2	5.2 E4	350	210	1.43	0,03	0,06	0.08	2.34	0.02	0.02	0.01	0.04	<dl	0.08	0.07	0.05	0.04	0.03	1E-2
R3	3.3 E4	130	110	0.15	28	0.03	0.07	0.55	0.01	6E-3	2E-3	0.02	1E-3	0.08	0.01	7E-3	0.01	6E-3	3E-3

A= Inside the plant – step Drying; B= Inside the plant – step Electromagnetic Separation; C= Inside the plant – step Gravimetric Separation; R1= residence number 1; R2= residence number 2; R3= residence number 3; dl=detection limit

Table 2. Mass Median Aerodynamic Diameter (MMAD) of airborne particles containing metals (μm).

	Elements																		
Local	**Cl**	**K**	**Ca**	**Ti**	**V**	**Cr**	**Mn**	**Fe**	**Co**	**Ni**	**Cu**	**Zn**	**Br**	**Zr**	**Ce**	**La**	**Pb**	**Th**	**Y**
A	-	-	1.7	1.7	-	1.5	1.4	1.6	-	1.6	-	2.1	-	2.2	-	-	1.6	1.7	-
B	-	-	1.5	1.4	-	1.3	1.0	1.3	-	1.5	1.5	1.7	-	1.4	-	-	1.4	1.4	-
	-	-	1.7	2.0	-	1.5	1.6	1.5	-	1.7	-	1.9	-	2.4	-	-	2.4	-	-
C	-	-	1.6	1.5	-	1.0	1.1	1.4	-	1.3	2.4	1.6	-	-	-	-	-	-	-
R1	1.6	1.6	1.5	1.6	2.2	2.2	1.9	1.2	0.7	1.7	1.9	1.7	1.9	2.2	1.8	1.9	2.0	1.2	1.8
R2	2.0	1.3	1.7	2.1	1.3	1.3	1.4	2.1	1.0	1.3	0.8	0.8	-	2.3	0.7	0.8	0.7	0.6	0.9
R3	1.1	0.8	1.0	0.7	0.7	1.0	0.8	0.7	1.0	0.7	0.8	0.8	0.8	2.1	1.0	1.1	0.8	1.1	1.1

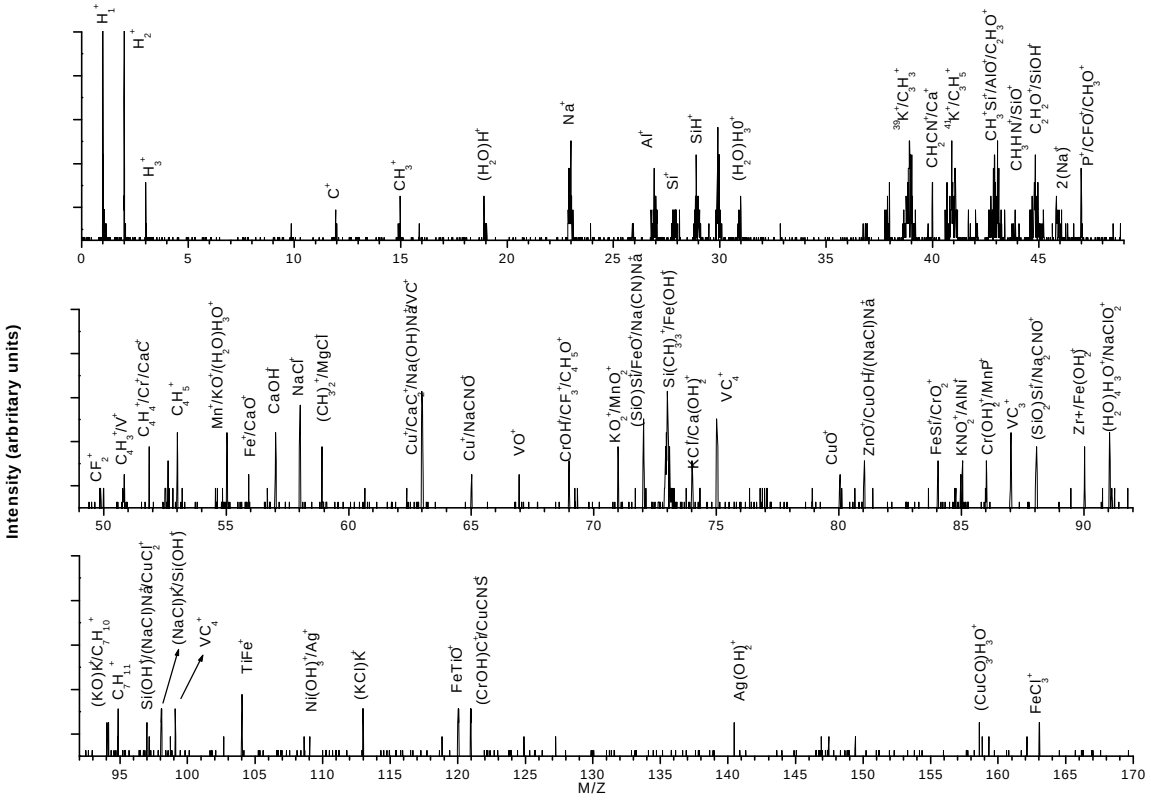
A= Inside the plant – step Drying; B= Inside the plant – step Electromagnetic Separation; C= Inside the plant – step Gravimetric Separation; R1= residence number 1; R2= residence number 2; R3= residence number 3

The mineral dust particles impacted on the 6th stage of the cascade impactor (particle with aerodynamic diameter in the range from 0.4 to 0.64 μm) during the electromagnetic separation process were analyzed by ^{252}Cf-PDMS. The positive spectra of mineral dust particle is shown in Figure 1. The mass spectrum of positive ions showed that Th was associated to oxygen (ThO_n) and silicon ($ThSiO_4$), indicating that thorite, not monazite, was the source of airborne particles containing thorium. The presence of cerium compounds was not observed in this mass spectrum, showing that there were not monazite dust particles in the respirable fraction of aerosol.

The ions of Ti and Fe observed in the mass spectra (^{252}Cf-PDMS method) showed that ilmenite ($TiFeO_2$) was the source of Ti and Fe bearing dust particles. The presence of zircon airborne dust particles ($ZrSiO.H_2O$) was also observed in the mass spectra.

The elements Ca, Cr, Mn, Ni, Cu, Zn and Pb observed in the X-ray and in the mass spectra indicate the existence of other sources of aerosol besides the mineral sands. Some of these sources could be the mechanical wearing of equipment, particles of oil used to lubricate or the burning fossil. Besides these sources, there were particles from soil (Al, Ni, Si, Zn, Fe and Ca) and marine aerosol (Cl, Ca, Na, K, Zn and Fe), which were transported.

Figure 1. Spectra of secondary ions obtained using ^{252}Cf-PDMS technique. Sample mineral dust particle.



The aerosol samples collected by AGF and cyclone were analyzed by alpha spectrometry and alpha and beta counter in order to determine the radionuclide concentration from natural series of thorium and uranium. Three consecutive samples were collected at the main entrance of the mineral sand plant. The ^{232}Th, ^{228}Th, ^{228}Ra, ^{238}U and ^{234}U concentrations in these aerosol samples were below the detection limits (0.8, 2.2, 0.9, 1.1 and 1.0 mBq/m^3 for each radionuclide, respectively). The average concentrations of ^{226}Ra in the fine and coarse fraction of the aerosol were 0.51 ± 0.18 and 0.43 ± 0.18 mBq/m^3 respectively. The ^{210}Pb coarse concentrations in the fine and coarse fraction of the aerosols were 0.48 ± 0.38 mBq/m3, and 0.38 ± 0.08 mBq/m^3, respectively. As the radionuclides present in the monazite are in secular equilibrium these results suggest that the ^{226}Ra and ^{210}Pb bearing particles could not be generated by the mineral process to concentrate monazite.

The ^{226}Ra and ^{210}Pb air concentrations in the samples collected using AGF and cyclone are below the air concentrations limit for these radionuclides adopted in Brazil [17].

Monazite is an orthophosphate of rare earths containing thorium and uranium associated to the crystal lattice. The thorium oxide concentration in monazite is about 6% and the uranium oxide concentration is 0.31% [18]. In the mineral sands plant, the four minerals (rutile, ilmenite, zircon and monazite) are separated by gravimetric and electro-magnetic processes and the monazite is not chemically processed at this plant. So, the secular equilibrium of the radionuclides observed in the monazite ore should be, also, observed in the aerosol samples. However, the ^{232}Th, ^{228}Th, ^{228}Ra, ^{238}U and ^{234}U concentrations were below the detection limit and only particles containing ^{226}Ra and ^{210}Pb (from the ^{238}U series) were observed in the aerosol samples. On the other hand, airborne particles containing Ce, La and Y were not observed, indicating that during the sampling the plant was not the source of radioactive particles in the fine fraction of aerosols.

The lichen, specimen Ramalina gracilis (Pres.) Nyl., samples were collected inside the exclusion area of the mineral sands processing plant (Buena village) and at the Manguinhos village. The samples collected at both villages were 15 years old and were used as bio indicators to characterize the presence of monazite dust during the last 15 years. The ^{232}Th, ^{228}Th, ^{228}Ra, ^{238}U and ^{234}U concentrations in the lichen samples are shown in Table 3. The secular equilibrium was observed among the radionuclides of the natural thorium series (^{232}Th, ^{228}Th and ^{228}Ra) in the lichen samples from Buena suggesting that the source of airborne particles containing these radionuclides were monazite dust particles. However, the ^{226}Ra and ^{210}Pb concentrations were higher than the ^{238}U and ^{234}U concentrations, indicating that the radionuclides from the natural uranium series were not in secular equilibrium, showing that the monazite was not the only source of radionuclides.

^{232}Th, ^{228}Th, ^{228}Ra, ^{238}U and ^{234}U concentrations in the lichen samples collected at the Manguinhos village were below the detection limits. The ^{226}Ra and ^{210}Pb concentrations in lichen samples from Manguinhos (Table 3) suggest that there was a source of ^{226}Ra and ^{210}Pb bearing particles in this village.

Table 3 ^{232}Th, ^{228}Th, ^{228}Ra, ^{238}U, ^{234}U and ^{210}Pb concentrations in lichen samples from Buena and Manguinhos village.

Radionuclides	Average Concentration (mBq/g)	
	Lichen sample location	
	Buena Village	Manguinhos Village
^{210}Pb	1810 ± 579	833 ± 61
^{226}Ra	1323 ± 396	849 ± 43
^{238}U	578 ± 24	< LC
^{234}U	556 ± 40	< LC
^{228}Ra	2165 ± 592	< LC
^{232}Th	1616 ± 395	< LC
^{228}Th	1317 ± 162	< LC

These results show that the inhabitants of the Buena village were, in the past, exposed to monazite dust particles. The concentrations of ^{226}Ra and ^{210}Pb bearing particles confirm that, besides the monazite processing plant there was another source of these radionuclides.

Buena and Manguinhos villages are located in a large swamp region. The ^{226}Ra and ^{210}Pb concentrations in swamp water were in the range of 0.06 to 0.63 and 0.008 to 0.1 Bq/L, respectively. However the U and

Th concentrations were below the detection limit (0.9 mBq/L) indicating that the swamp could be a source of ^{226}Ra and ^{210}Pb bearing particles [18].

The airborne particles in the fine fraction of aerosols have been generated by evaporation and wind transport processes on the water surface of the swamp. The results of the lichen samples and of the aerosol characterization suggest that the swamp could be the source of ^{226}Ra and ^{210}Pb bearing particles besides the monazite.

The ^{232}Th average concentration in 24 h urine samples from the Buena village inhabitant was 1.8 mBq/L [3]. This sample was analyzed by ^{232}Cf-PDMS. The ions present in the human urine (Na^+, Mg^+ and Ca^+), $NaCl^+$, $CaC_2O_4^+$ and the main organic compounds were identified in the mass spectrum of positive ions. The phosphates, sulphates, NHO_3^-, $C_5H_{11}O_5^-$ normally present in the human urine and the CN clusters, that characterize the organic compounds, were also observed in the negative ions spectrum. The urine mass spectra were compared to the mass spectrum of airborne particles collected during the ilmenite concentration [14,15] and to the mass spectrum of a sample of concentrate of monazite [15]. The $FeTi^+$ ions were identified in the mass spectra of some the of urine samples, indicating the presence of ilmenite. The identification of these molecular ions in the urine mass spectra shows that the inhabitants were exposed to ilmenite particles by inhalation, being probably part of ilmenite incorporated not dissolved by physiological processes. The Zr^+ and ZrO^+ were also identified in the spectra suggest that the inhabitant incorporate zirconite. The La^+, Ce^+, $LaPO_3^-$ and $LaPO_4^-$ were identified in the urine sample of the Buena inhabitant. The presence of yttrium oxide in the urine mass spectrum suggests that part of the yttrium incorporated is not metabolized. The thorium compound identified in the positive mass spectrum was $ThNH_4$, showing that the thorium present in urine was the product of thorium compound metabolized by the human body. The urine mass spectra suggest that thorium could be associated to a protein before it goes to urinary tract. The identification of La, Ce, Y and Th compounds in urine mass spectra shows that the inhabitant has incorporated the monazite by inhalation.

The mass spectra from inhabitant of Buena village were compared to mass spectra of one individual never exposed to monazite particulate (inhabitant of Rio de Janeiro city). The lines characterizing the monazite (La, Ce, Y and Th) were not identified in the mass spectrum of control.

Conclusions

The airborne particles characterization suggests that inhabitants of Buena village and workers in the mineral processing plant are exposed to mineral sands particles, marine aerosols and particles from anthropogenic sources in the respirable fraction of aerosols. The sources of mineral sands particles are the mineral plant and natural deposit of mineral sands located in the village. The ^{252}Cf-PDMS results showed that although the plant processes mineral sands, the source of thorium bearing particles was thorite sand present in the soil of the region from which monazite is extracted. The ilemenite, zircon and thorite particles were identified in the fine fraction of aerosol. Based on the PIXE and PDMS analysis it was possible to conclude that there were other sources of aerosol, which produce airborne dust particles containing Th in the respirable fraction.

The mass spectra of the urine sample from a inhabitant of Buena village and of Rio de Janeiro city show the lines of molecules ions that characterize main compounds present in the human urine (NH_3, NaCl, oxalate acid, K, Mg, etc). The lines of TiFe, La, Ce, Zr, Y and $ThNH_4$ in the urine sample from the Buena inhabitant characterize metal incorporation probably by inhalation. These results suggest that the main source of metal in Buena village is the minerals sands and that the inhabitant inhales monazite particulate.

^{232}Th, ^{228}Th, ^{228}Ra, ^{238}U and ^{234}U were not identified in the aerosol, characterizing that the inhabitant were not exposed to monazite particles during the period of sampling. The fact that the secular equilibrium of

the radionuclides from the natural series of Th and U was not observed in the samples indicates that there is another source of ^{226}Ra and ^{210}Pb, besides the mineral sands processing plant, in the village.

The main anthropogenic sources of particles are the mineral sands processing plant and the truck traffic and the natural sources are the sea, soil and the swamp.

Based on the results from the lichen samples it could be concluded that at least during the last 15 years the inhabitants of the village have been exposed to monazite particles. The results suggest that the swamp was also a source of ^{226}Ra and ^{210}Pb bearing particles besides the monazite dust.

The elemental mass and activities concentrations determined in the present study were below the Brazilian limits suggesting that although the inhabitants of Buena village are exposed to different source of aerosols there is no acute damage for their health. However, the risk due a chronic exposure to low-level should be investigated.

Results of urine sample from Buena by ^{252}Cf-PDMS technique agree with the results of aerosol characterization. These results suggest that thorium was metabolized and probably has been associated to a protein in the human body. However metals such as Y could be not metabolized and eliminated as oxide in urine. The employment of PIXE and ^{252}Cf-PDMS techniques for the analysis of environmental and biological samples is an important tool for characterization of human and metal contamination. The knowledge of this information can provide the base for the identification of the sources of pollution and permit evaluation of the environment impact due to the industrial.

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