

INVENTORY OF ^{226}Ra , ^{228}Ra AND ^{210}Pb IN MARINE SEDIMENTS CORES OF SOUTHWEST ATLANTIC OCEAN

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ABSTRACT

^{210}Pb (22.3 y) is a radioactive isotope successfully applied as tracer of sediment dating of the last 100-150 years. The application of ^{226}Ra and ^{228}Ra as paleoceanographic tracers (half-lives of 1,600 y and 5.7 y, respectively) also gives some information of ocean's role in past climate change. In this work, it was analyzed 2 sediment cores collect at Southwest Atlantic Ocean. The sediments samples were freeze-dried and acid digested in microwave. It was carried out a radiochemical separation of ^{226}Ra , ^{228}Ra and ^{210}Pb and performed a gross alpha and gross beta measurement of both precipitates $\text{Ba}(\text{Ra})\text{SO}_4$ and PbCrO_4 in a low background gas-flow proportional counter. Activity concentrations of ^{226}Ra ranged from 45 Bq kg^{-1} to 70 Bq kg^{-1} in NAP-62 and from 57 Bq kg^{-1} to 82 Bq kg^{-1} in NAP-63 samples. The concentration of ^{228}Ra varied between 37 Bq kg^{-1} and 150 Bq kg^{-1} in NAP-62 and between 23 Bq kg^{-1} and 111 Bq kg^{-1} in NAP-63 samples. The concentration of total ^{210}Pb ranged from 126 Bq kg^{-1} to 256 Bq kg^{-1} in NAP-62 and from 63 Bq kg^{-1} to 945 Bq kg^{-1} in NAP-63 samples. Results of $^{210}\text{Pb}_{\text{uns}}$ varied from 68 Bq kg^{-1} to 192 Bq kg^{-1} for NAP-62, while varied from <4.9 Bq kg^{-1} to 870 Bq kg^{-1} in NAP-63 profile. Increased values of $^{210}\text{Pb}_{\text{uns}}$ were found on the top of both NAP-62 and NAP-63 sediment profile.

1. INTRODUCTION

The continental platform is an interaction area between marine environment, continent and atmosphere. The Southeast Brazilian Continental Platform extends up to the 180 meters isobath and it's a place where many geological data is available, most of them related to actual overlying seabed sediment. Natural radioactive isotopes have been successfully applied as tracers of environmental changes occurred in the past or in the actual time. The seabed sediment of the Southeast Brazilian Continental Platform is basically formed of continental material [1-3].

The geochemical study of marine sediments is a very important source of information about the past environment. Within many kinds of measures, the U and Th series radionuclides stand out by their capability to generate timescales of different environmental processes (Fig.1).

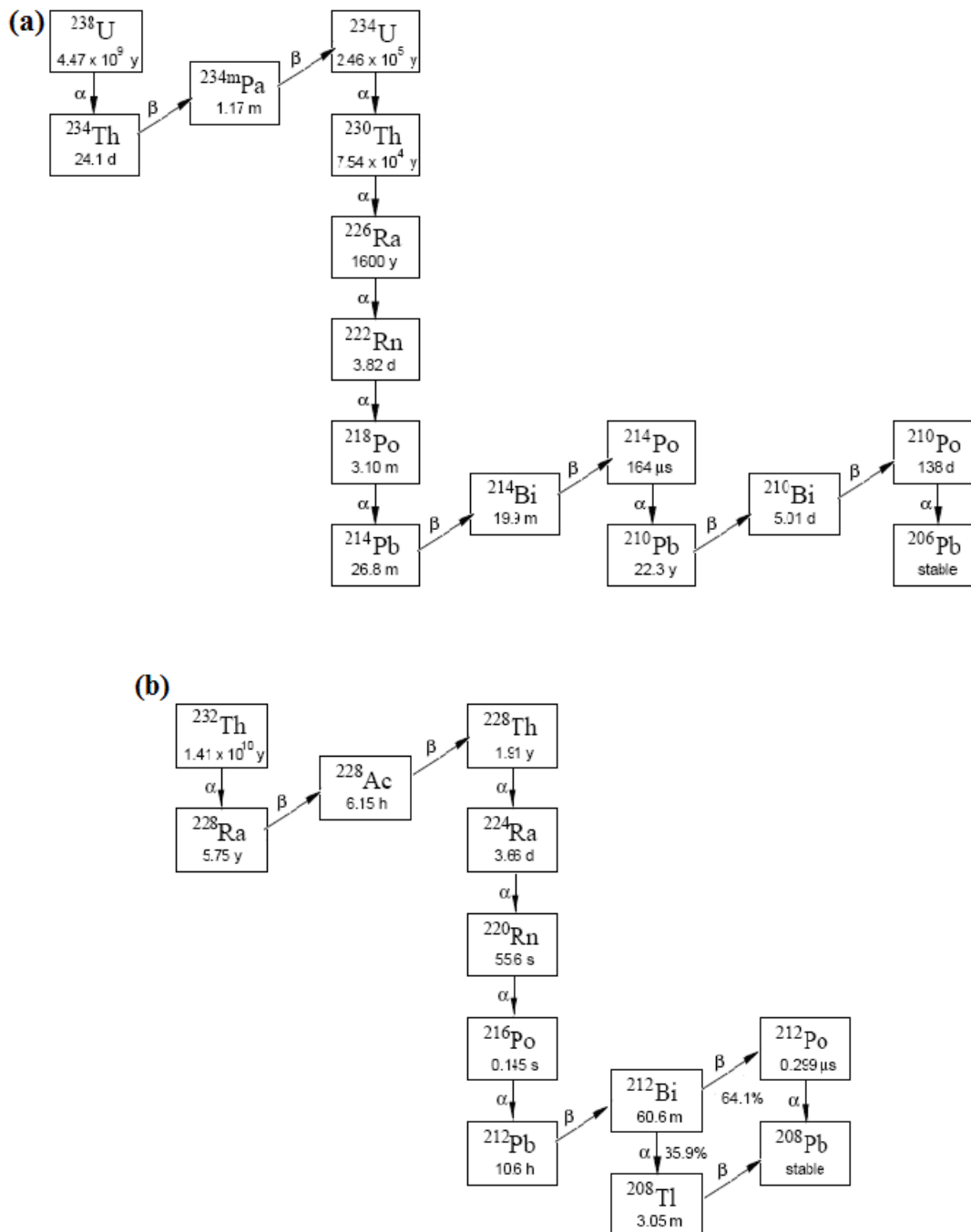


Figure 1: Decay chain of ^{238}U (a) and ^{232}Th (b).

The Pb element is insoluble in seawater. At the seawater column, it can be adhered at sinking particles until it reaches the bottom of the sea. The mainly Pb isotope found in the sea is the ^{210}Pb (^{226}Ra and ^{222}Rn daughter). The isotope ^{210}Pb is found at the atmosphere as a result of a

sequence of events: the ^{226}Ra , present in the earth's crust, decay to ^{222}Rn (gas) which emanates from the minerals to the atmosphere. The atmospheric ^{222}Rn decays to ^{210}Pb which in a short time get together with atmospheric particles and return to the earth's surface by the atmospheric precipitation as rain and snow. The ^{210}Pb present in the top of the core is found in excess when compared with its precursor, ^{226}Ra , generating the ^{210}Pb not supported. The not escaping ^{222}Rn decays to ^{210}Pb and reach the radioactive equilibrium with the ^{226}Ra , generating the ^{210}Pb supported (Fig.2). The depth and penetration pattern of ^{210}Pb (half-life of 22.3 years) through the sediment is a well known technique of sediment dating of the last 100-150 years and the determination of sedimentation rates. Due to ^{210}Pb half-life, its activities in the sediment profiles have an exponential decrease through the depth [4].

The Ra element is soluble in seawater. The ^{226}Ra sources in seawater come from the ^{238}U dissolved and also derives in part of ^{230}Th decay in marine sediments. The ^{228}Ra has its growth given by the decay of ^{232}Th , also rather found in the seabed sediment. After the respectively precursor decay, both ^{226}Ra and ^{228}Ra are release to the seawater in soluble forms (Fig. 2). The study of ^{226}Ra and ^{228}Ra concentrations (half-lives of 1,600 years and 5.7 years, respectively) also can give some information of the ocean's role in past climate change.

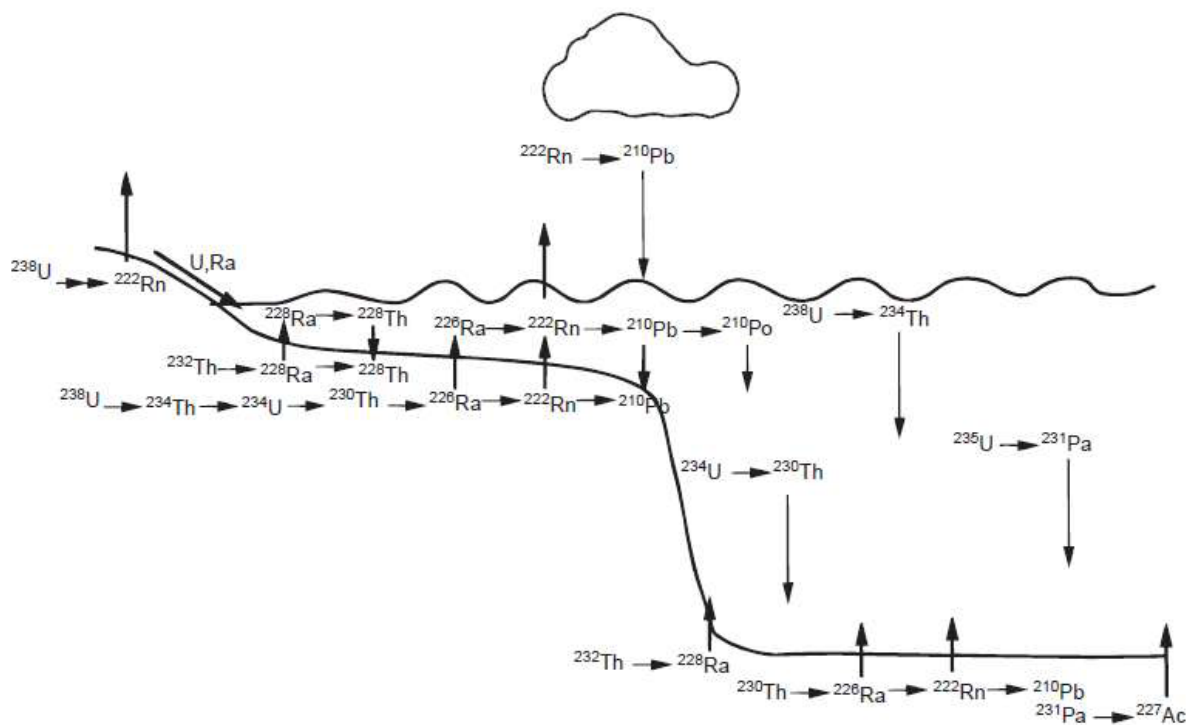


Figure 2: Schematic diagram of natural radioactive decay and its transport process in the ocean and atmosphere [5].

The northernmost part of the southern Brazilian margin, formerly known as the São Paulo Bight, is an arc shaped embayment extending from 23°S to 28°S. The hydrodynamic control, together with the relative tectonic stability and the absence of post-glacial rebound, makes the area a favorable site for investigations of the Late Quaternary climatic changes of the southwestern Atlantic. The area has a big potential for studying changes related to the Last

Climatic Cycle, sub-Milankovitch variations such as latitudinal shifts of the Intertropical Convergence zone (ITCZ) and variations in the El Niño Southern Oscillation (ENSO) [6].

On the other hand, due to the flourishing oil and gas exploration activities and related engineering and environmental aspects such as the installation of pipeline sand platforms, studies on sedimentary processes are of great importance.

The objective of this work is to develop an inventory the ^{210}Pb , ^{226}Ra and ^{228}Ra isotopes in sediments of the São Paulo Bight (southern Brazilian margin) and to analyze the data in terms of Late Quaternary sedimentary process and sedimentation rates.

2. MATERIALS AND METHODS

2.1. Study Area

The ocean floor of the São Paulo Bight shows a rather complex morphology. The shelf break is located at water depth of approximately 140 meters, with the upper slope showing an average gradient of about 1:55 [7]. The distribution of surface sediments on the southeastern Brazilian margin was extensively studied and in general, the present sea floor is covered by very fine siliciclastic sands and silts with variable amounts of clay and calcium carbonate. Coarser terrigenous sediments, carbonate gravel and boulder facies found on the outer shelf represent less than 5% of the present bottom, generally related to relict sediments that are deposited under lower sea level conditions.

Sedimentary processes in the area have been reevaluated in terms of the controlling hydrodynamic processes. On the inner shelf, the sedimentation is mainly determined by the displacement of the Brazilian Coastal Current (BCC) [8]. On the middle and outer shelves, as well as on the upper slope, the sedimentary processes are mainly influenced by the southward flow of the Brazil Current (BC) along the western Atlantic continental margin [9-10].

For the purposes of this research work, 2 short sediment corers were collected using a multiple-corer (MUC). The sampling cruise was carried out in February, 2013 onboard of the N/Oc. Alpha Crucis, belonging to the Oceanographic Institute of the University of São Paulo (IO/ USP). The study was performed as a part of the project “Inner Shelf Sedimentation Patterns of the Southeast Brazilian Continental Platform: base for paleoclimatic models, paleoceanography, oil and gas exploration, Subproject-5 (NAP GEO-SEDEX)”.

The NAP-62 and NAP-63 sediment sampling stations are shown in the Fig. 3. In Table 1 are presented the geographic coordinates and water column depths of each station and the overall recuperation of each sediment core. After sampling and sediment stratigraphic analysis, both cores were sliced in 1 cm intervals and radiochemically analyzed at 4 cm intervals.

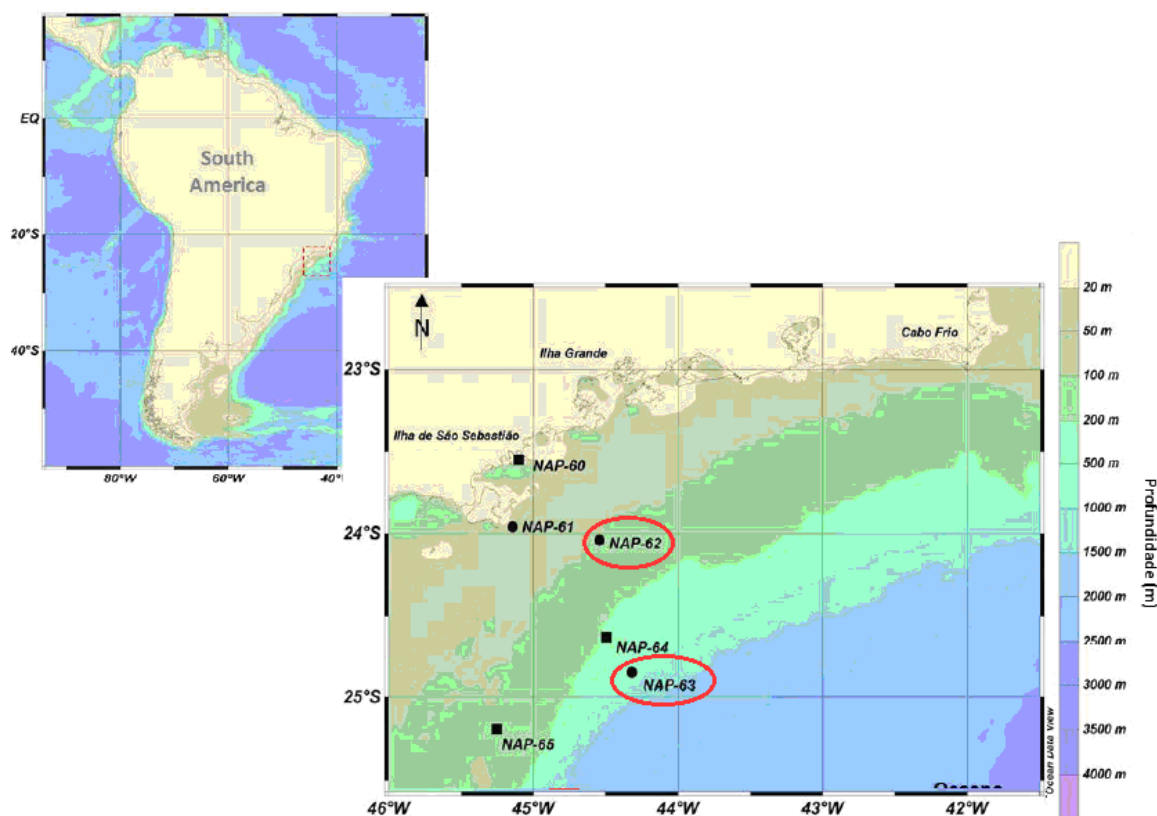


Figure 3: Map of São Paulo Bight. The red circles indicate NAP-62 and NAP-63 sediment sampling stations.

Table 1: Geographical coordinates and depths of each oceanographic station, and the respectively core recuperation.

Station	Equipment used	Latitude (°S)	Longitude (°W)	Depth of water column (m)	Core lenght (cm)
NAP-62	MUC	24.042	44.542	121	28
NAP-63	MUC	24.840	44.319	840	35

2.2. Sequential Radiochemical Separation of ^{226}Ra , ^{228}Ra and ^{210}Pb

After sediment sampling, the removal aliquots for testing was taken every 4 cm. These aliquots were stored in labeled polyethylene flasks with its respective information. The samples were freeze-dried and subsequently submitted through the homogenization process.

As for the sequential radiochemical determination of ^{226}Ra , ^{228}Ra , and ^{210}Pb in the sediment, aliquot samples were obtained by weighting in an analytical balance, with masses around 0.5 g. These samples were transferred to a Teflon tube for future solubilization in a microwave digester. Samples were analyzed in replicates. The methodology is suitable to quantify these radionuclides in small amounts of marine sediments in which they are present mostly in low concentrations or as traces. The chosen technique has as positive the low background radiation and thus a considerably lower limit of detection.

Following the sample homogenization, each sample was passed through an acid leaching method, which provides the solubilization according to the EPA-3052 protocol [11]. This protocol is applicable to assisted digestion of sediment matrices in a microwave, with acidic digestion pressurized pipe. The acid combination chosen used a ratio 3:1 nitric acid (HNO₃), hydrofluoric acid (HF) and hydrochloric acid (HCl). This is suitable for stabilizing high concentrations of Fe, Al, Ag, Ba and Sr in solubilized samples. The total sample digestion was required for decomposition of the sedimentary matrix and stabilization of specific elements in high temperature reactions (EPA-3052), with the help of brand digester CEM microwave, MARS-6 model. All the acids used were concentrated with high purity to minimize interferences in the samples, and the use of hydrogen peroxide (H₂O₂) (30%) helped in the elimination of organic matter [11]. Aliquots of 20 ml obtained after the leaching process of the samples were filtered and diluted to 1 L with Milli-Q purified water to initiate the radiochemical procedure.

The sequential radiochemical separation of ²²⁶Ra, ²²⁸Ra, and ²¹⁰Pb was performed using the methodologies described in Oliveira (1993) [12] and Moreira (1993) [13] (Fig. 4). Carriers of stable barium (20 mg) and lead (20 mg) were added to the samples in the presence of 5 mL of 1M citric acid and 5 mL of 40% hydroxylamine hydrochloride solutions.

The Ra and Pb were co-precipitated as Ba(Ra,Pb)SO₄ by adding 50 mL of 3 M H₂SO₄. The precipitate was dissolved with alkaline EDTA. When the pH is adjusted to 4.5 with glacial acetic acid, Ba(Ra)SO₄ is re-precipitated, while interfering elements and Pb remains in the solution. The Pb is, in turn, precipitated as PbCrO₄ by the addition of Na₂CrO₄.

After a repose time of 10 days for ²¹⁰Pb and 21 days for ²²⁶Ra and ²²⁸Ra, both precipitates were measured by gross alpha and gross beta in a low background gas-flow proportional counter, model Berthold LB-770, from EG&G Berthold. The counting time was defined of 200 min. The chemical yield was calculated gravimetrically by the initial addition of Ba²⁺ and Pb²⁺ carrier solutions. The experimental radiochemical procedure was carried out in the Environmental Radiometrics Laboratory (LRA) of IPEN.

The chemical yield was determined gravimetrically by weighing the final mass of Ba(Ra)SO₄ and PbCrO₄ precipitates, considering the initial additions of 20 mg of Ba²⁺ and Pb²⁺ carrier solutions, respectively. As for the Ra isotopes determination, 100% chemical yield corresponded to a mass of 34.1 mg of Ba(Ra)SO₄. For the radiochemical separation of Pb, 100% chemical yield matches a mass of 31.4 mg of PbCrO₄. The percentual mean chemical yield obtained for the Ba(Ra)SO₄ was 89 ± 8 %, and for the PbCrO₄ was 80 ± 10%.

Typical lower limits of the detection (LLD) for these methods were 2.2 Bq kg⁻¹ for ²²⁶Ra, 3.7 Bq kg⁻¹ for ²²⁸Ra, and 4.9 Bq kg⁻¹ for ²¹⁰Pb, at a 95% confidence level. These limits were estimated considering an initial sediment aliquot of 0.5 g per sample and a counting time of 200 minutes.

^{226}Ra , ^{228}Ra and ^{210}Pb radiochemical separation procedure:

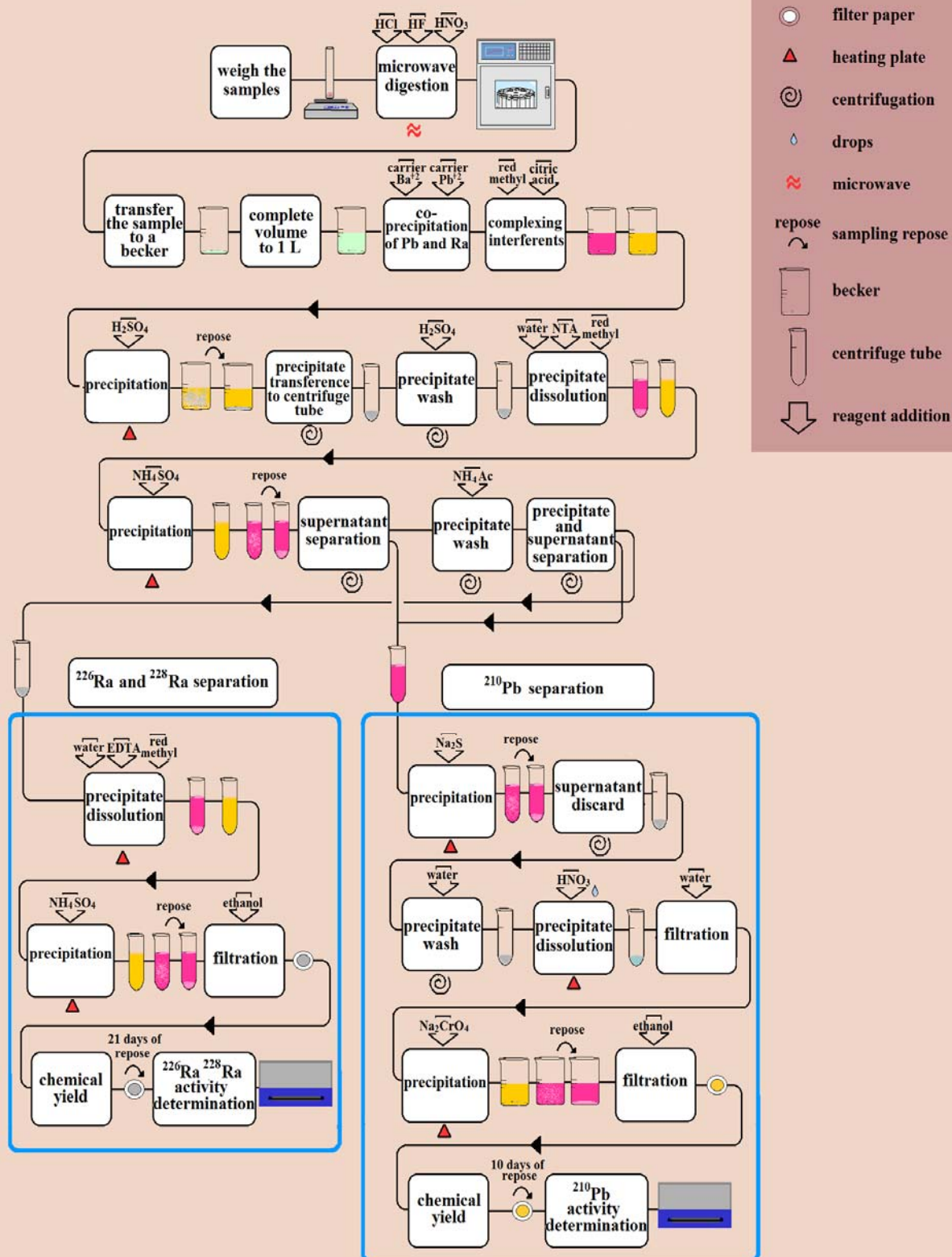


Figure 4: Flowchart of the ^{226}Ra , ^{228}Ra and ^{210}Pb radiochemical separation procedure.

3. RESULTS AND DISCUSSION

The arithmetic mean activity concentrations of ^{226}Ra , ^{228}Ra , total ^{210}Pb and ^{210}Pb unsupported determined in sediment profile NAP-62 are presented in Table 2. Activity concentrations of ^{226}Ra ranged of 45 Bq kg^{-1} to 70 Bq kg^{-1} ; for ^{228}Ra activities varied from 37 Bq kg^{-1} to 150 Bq kg^{-1} . The activity concentration of total ^{210}Pb was in the interval of 126 Bq kg^{-1} to 256 Bq kg^{-1} , while activities of $^{210}\text{Pb}_{\text{uns}}$ ranged from 68 Bq kg^{-1} to 192 Bq kg^{-1} .

According to the data showed in Table 2, it was observed a very small variation in the activity concentrations of ^{226}Ra along the sediment column NAP-62. The activities of ^{228}Ra in sediment profile NAP-62 presented in Table 2 show an increase of the concentration at 25 cm depth, which probably reflects sediment remobilization and/ or bioturbation processes. Characteristics of reworking and/or bioturbation in sediment cores of the same area was also seen in Mahiques et al. (2011) [14].

It is important to note that NAP-62 is a sediment sample taken closer to the continent than NAP-63 that means possibly more subjected to the overall remobilization processes. It should be taking into account however that the mixture of the first sediment layers by benthonic animals, in the most of the time, can limit the paleoceanographic data resolution of NAP-62, given its lower collecting depth.

In the case of NAP-62 sediment profile, the highest activity concentrations of $^{210}\text{Pb}_{\text{uns}}$ were observed from 0-9 cm depth, indicating the main contribution of ^{210}Pb from atmospheric deposition.

Table 2: Arithmetic mean activity concentrations of ^{226}Ra , ^{228}Ra , $^{210}\text{Pb}_{\text{total}}$ and $^{210}\text{Pb}_{\text{uns}}$ of the sediment profile NAP-62.

Sample	$A^{226}\text{Ra}$ (Bq kg^{-1})			$A^{228}\text{Ra}$ (Bq kg^{-1})			$A^{210}\text{Pb}_{\text{total}}$ (Bq kg^{-1})			$A^{210}\text{Pb}_{\text{uns}}$ (Bq kg^{-1})		
[NAP62 #0-1 cm]	64	±	6	67	±	13	191	±	25	127	±	17
[NAP62 #4-5 cm]	63	±	6	65	±	13	249	±	30	186	±	23
[NAP62 #8-9 cm]	64	±	6	99	±	10	256	±	51	192	±	38
[NAP62 #12-13 cm]	70	±	7	37	±	7	142	±	14	72	±	7
[NAP62 #16-17 cm]	57	±	6	64	±	13	183	±	37	126	±	25
[NAP62 #20-21 cm]	58	±	6	49	±	10	126	±	25	68	±	14
[NAP62 #24-25 cm]	45	±	5	150	±	30	ND			ND		
[NAP62 #27-28 cm]	ND			ND			ND			ND		

Legend:

LLD (lower limit of the detection): $^{226}\text{Ra} = 2.2 \text{ mBq g}^{-1}$, $^{228}\text{Ra} = 3.7 \text{ mBq g}^{-1}$, $^{210}\text{Pb} = 4.9 \text{ mBq g}^{-1}$

ND (not determined); $^{210}\text{Pb}_{\text{uns}} = ^{210}\text{Pb}_{\text{total}} - ^{226}\text{Ra}$

In the same manner, the arithmetic mean activity concentrations of ^{226}Ra , ^{228}Ra , total ^{210}Pb and ^{210}Pb unsupported estimated for sediment profile NAP-63 are presented in Table 3. Activity concentrations of ^{226}Ra ranged of 57 Bq kg^{-1} to 82 Bq kg^{-1} ; for ^{228}Ra activities varied from 23 Bq kg^{-1} to 111 Bq kg^{-1} . The activity concentration of total ^{210}Pb was in the interval

of 63 Bq kg⁻¹ to 945 Bq kg⁻¹, while activities of ²¹⁰Pb_{uns} ranged from < 4.9 Bq kg⁻¹ to 870 Bq kg⁻¹.

The distribution of ²²⁶Ra activity concentrations is almost constant along NAP-63 profile. As for as ²²⁸Ra activity concentrations, it was observed increased values from 8 cm to 12 cm depth, and for 20 cm and 32 cm depth. The highest values of total ²¹⁰Pb and ²¹⁰Pb_{uns} were found in the top of NAP-63 (Table 3).

Table 3: Arithmetic mean activity concentrations of ²²⁶Ra, ²²⁸Ra, ²¹⁰Pb_{total} and ²¹⁰Pb_{uns} of the sediment profile NAP-63.

Sample	A ²²⁶ Ra (Bq kg ⁻¹)	A ²²⁸ Ra (Bq kg ⁻¹)	A ²¹⁰ Pb _{total} (Bq kg ⁻¹)	A ²¹⁰ Pb _{uns} (Bq kg ⁻¹)
[NAP63 #0-1 cm]	75 ± 7	53 ± 11	945 ± 79	870 ± 8
[NAP63 #4-5 cm]	60 ± 6	51 ± 10	134 ± 27	74 ± 15
[NAP63 #8-9 cm]	58 ± 6	94 ± 19	149 ± 30	91 ± 18
[NAP63 #12-13 cm]	82 ± 7	103 ± 20	90 ± 18	8 ± 2
[NAP63 #16-17 cm]	81 ± 8	72 ± 14	75 ± 15	< 4.9
[NAP63 #20-21 cm]	63 ± 6	111 ± 22	ND	ND
[NAP63 #24-25 cm]	68 ± 4	72 ± 14	63 ± 13	< 4.9
[NAP63 #28-29 cm]	57 ± 6	23 ± 5	128 ± 26	71 ± 14
[NAP63 #32-33 cm]	66 ± 7	82 ± 17	110 ± 22	44 ± 9
[NAP63 #34-35 cm]	ND	ND	108 ± 22	ND

Legend:

LLD (lower limit of the detection): ²²⁶Ra = 2.2 mBq g⁻¹, ²²⁸Ra = 3.7 mBq g⁻¹, ²¹⁰Pb = 4.9 mBq g⁻¹

ND (not determined); ²¹⁰Pb_{uns} = ²¹⁰Pb_{total} - ²²⁶Ra

The data collected in this research work was evaluated by comparison with others relative to ²²⁶Ra, ²²⁸Ra, total ²¹⁰Pb and ²¹⁰Pb_{uns} distributions in Southwest Atlantic Ocean, available in the literature.

The activity concentrations of ²²⁶Ra, ²²⁸Ra, total ²¹⁰Pb and ²¹⁰Pb_{uns} in this work were lower than the values determined in Morrão River estuary, Brazil, in the proximity of Cubatão and Santos City, reaching the magnitude found in estuaries with strong anthropogenic influence, mainly affected by fertilizer industries [15]. These high values of ²¹⁰Pb compared to ²²⁶Ra were related to important releases of phosphogypsum into an estuary or to ²²⁶Ra remobilization from anaerobic coastal sediments. In this work activity the following activity concentrations ranges were reported: ²²⁶Ra (142-744 Bq kg⁻¹), ²¹⁰Pb_{total} (341-1305 Bq kg⁻¹) and ²¹⁰Pb_{total} (100-700 Bq kg⁻¹) are recorded in 1 sediment core of 30 cm length.

Non contaminated sediments of Southeast Brazilian Coast were measured by many other authors and it was obtained values between 2 Bq kg⁻¹ to 29 Bq kg⁻¹ for ²²⁶Ra, between 14 Bq kg⁻¹ to 192 Bq kg⁻¹ for ²¹⁰Pb and between 0,05 Bq kg⁻¹ to 163 Bq kg⁻¹ for ²¹⁰Pb_{uns} [16-20]. The only one that performed ²²⁸Ra analyzes [18] found values between <19 Bq kg⁻¹ to 55 Bq kg⁻¹. The data collected in this paper are in the same order of magnitude of those of non-contaminated areas.

The research made in 1 sediment core collect in Guanabara Bay (70 cm) [16], a highly eutrophicated area, found some low activity concentration levels at the depth of 1-2 cm which was explained by the bioturbation, by the beginning of the anoxic zone or by compactation.

Saito *et al.* (2000) [17] analyzed 4 sediment cores between 34-44 cm of length in Cananéia-Iguape Estuary, Brazil and concluded that the influence of sediment sources were related to small rivers and the sediment resuspension caused by tide currents. Some low radionuclide levels were correlated with the sediment granulometry.

The research which evaluated the environmental impact from discharges of ^{226}Ra and ^{228}Ra by produced water from Bacia de Campos, Brazil, oil field offshore platforms, [18] concluded that, in spite of the elevated concentrations of radium in produced water from the oil field, the obtained concentrations in the sediment samples are at the local background level.

The study of 1 sediment core of 40 cm length of Guaratuba Bay, Brazil [19] generated an almost linear decline of ^{210}Pb total activity in relation to depth and a consistent behavior of ^{226}Ra activity throughout the sediment core.

Wanderley *et al.* (2014) [20] studied 14 sediment cores of 4 transects collected in the Paraíba do Sul estuary and its shelf regions. The pattern of an almost constant concentration of ^{210}Pb with depth in some sediment cores was related to anthropogenic activities, such as trawling, which is common in the region, bioturbation by abundant polychaetes or even erosional processes. The occurrence of ^{210}Pb peaks was observed in several sediment cores which could indicate a variable concentration of ^{226}Ra along the core, invalidating the subtraction of fixed ^{210}Pb values for all layers. Differences in sedimentation rates may result from land-use changes in the drainage basin, hydrologic variations, and natural and erosional events.

3. CONCLUSIONS

The activity concentration levels of the natural radionuclides ^{226}Ra , ^{228}Ra , total ^{210}Pb and $^{210}\text{Pb}_{\text{uns}}$ were studied in 2 short sediment cores (NAP-62 and NAP-63) collected at the Southwest Atlantic Ocean in February/ 2013. Until up today, there is still a lack of such data regarding activity distributions of these radionuclides in sediments, when compared with studies performed in North Atlantic and Pacific Oceans. Mapping these radionuclides in Brazilian Southwest Atlantic is relevant to better understand mechanisms of sedimentation transport and accumulation, besides to contribute for the development of paleoceanographic and geochronological studies.

The ^{226}Ra , ^{228}Ra , total ^{210}Pb and $^{210}\text{Pb}_{\text{uns}}$ inventory was determined by using the gross alpha and gross beta measurements in a low background proportional counter, after a sequential radiochemical separation. Activity concentrations of ^{226}Ra ranged from 45 Bq kg⁻¹ to 70 Bq kg⁻¹ in NAP-62 and from 57 Bq kg⁻¹ to 82 Bq kg⁻¹ in NAP-63 samples. The concentration of ^{228}Ra varied between 37 Bq kg⁻¹ and 150 Bq kg⁻¹ in NAP-62 and between 23 Bq kg⁻¹ and 111 Bq kg⁻¹ in NAP-63 samples. The concentration of total ^{210}Pb ranged from 126 Bq kg⁻¹ to 256 Bq kg⁻¹ in NAP-62 and from 63 Bq kg⁻¹ to 945 Bq kg⁻¹ in NAP-63 samples. Results of $^{210}\text{Pb}_{\text{uns}}$ varied from 68 Bq kg⁻¹ to 192 Bq kg⁻¹ for NAP-62, while varied

from $<4.9 \text{ Bq kg}^{-1}$ to 870 Bq kg^{-1} in NAP-63 profile. Increased values of $^{210}\text{Pb}_{\text{uns}}$ were found on the top of both NAP-62 and NAP-63 sediment profile.

The results presented in this paper are still in progress and other 2 sediment cores from the area are under analysis. Future work intends to apply all the data in geochronological dating models, in order to evaluate sedimentation rates.

ACKNOWLEDGMENTS

I would like to thank my advisor's help, Dr. Joselene de Oliveira; the support of Dr. Rubens Cesar Lopes Figueira, who provided the sediment samples; the laboratory colleagues; and "Comissão Nacional de Energia Nuclear (CNEN)" for provide a Master in Science scholarship.

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