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Neutron activation analysis applied in sediment samples from the Guarapiranga Reservoir for metals and trace elements assessment

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Abstract The Guarapiranga Reservoir is a very important aquatic system due to the fact that it is one of the main water reservoirs for South America's largest city, São Paulo, Brazil. Guarapiranga basin is located within the Metropolitan Region of São Paulo and the reservoir itself is located in the Northern part of the basin occupying approximately 26 km^2. This reservoir is characterized by environmental impacts from urban invasion, industrial and sewage wastes, all of which seriously affect its water quality and, consequently, the sediment quality. Two collection campaigns were undertaken: April 2009 and June 2010. The samples were analyzed by instrumental neutron activation analysis (INAA) in order to determine the following elements: major (Fe, K and Na), trace (As, Ba, Br, Co, Cr, Cs, Hf, Rb, Sb, Sc, Ta, Tb, Th, U and Zn) and rare earths (Ce, Eu, La, Lu, Nd, Sm, Tb and Yb). The organic matter and granulometric distribution were also evaluated and multivariate analysis was applied to the results. The study of elemental ratios indicated that the amount of elements present in the Guarapiranga Reservoir is mainly of detrital origin.

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Introduction

The Metropolitan Region of São Paulo, Brazil, represents the largest and most populous urban area in South America, with an area of almost 8,000 km^2. Water for the Metropolitan Region of São Paulo is supplied by a set of eight water production systems, treatment and distribution [1–3]. Water sources are surface sources or groundwater used for human consumption and maintenance of economic activity [4], and Guarapiranga Reservoir is one of the main water sources of the Metropolitan Region of São Paulo.

The Guarapiranga Dam is a sub-basin of the Upper Tietê Basin and is located fully in the State of São Paulo, extends over an area of approximately 63,911 hectares (639.11 km^2) and is responsible for supplying enough water for about 3.7 million people—20% of the population of the Metropolitan Region of Sao Paulo [1, 5, 6].

In 1903, Sao Paulo faced a crisis in its water supply due to the prolonged dry season and in 1906 began the construction of the dam that originated the Guarapiranga Reservoir and finished in 1909. In the 1970s, urban centers began to settle in the region without the necessary infrastructure of basic sanitation for the population and, at the end of 1980s, creating environmental impacts due to the large amount of untreated sewage discharged directly into the waters of the Reservoir [3, 5].

The sediment is a complex array of aquatic ecosystems, because it is responsible for the transport, release and accumulation of toxic compounds and nutrients, and may represent a source of metal contamination of the aquatic environment. Sediment is the layer of particulate material found at the bottom of natural water bodies, being the result of continuous processes of weathering and erosion of rock and mineral surfaces [7, 8]. Therefore, the sediment is an important compartment for the study of aquatic ecosystems contamination and their chemical characterization is necessary for the understanding of natural processes and human influence on these proceedings [9].

The Environmental Company of São Paulo State (CETESB) is responsible for environmental monitoring in the entire State of Sao Paulo, especially natural bodies of water. CETESB has one sediment monitoring point in the reservoir, where the collection is made once a year for metals and other parameters evaluation. The objective of this study was to characterize the sediments of the Guarapiranga Reservoir through the assessment of the concentrations of metals and trace elements present in samples collected throughout the reservoir and to understand their geochemical correlations by using multivariate analysis tools. The data of this study can be used also a complement to the CETESB data since this agency has data concerning one collection per year and only at one point, as mentioned above.

Materials and methods

Sampling and sample preparation

Two sampling campaigns (April 2009 and June 2010) were carried out and five bottom sediment samples (SG01 to SG05) were collected in the Guarapiranga Reservoir by a vanVeen sampler (Fig. 1). The geographical positions and other details of the sampling points are presented in Table 1. Sediment samples were previously dried at 45 °C in a ventilated oven until constant weight, passed through a 2 mm sieve, ground in a mortar, once again passed through a 75 μm sieve and then homogenized before analysis. The total fraction (<2 mm) was analyzed.

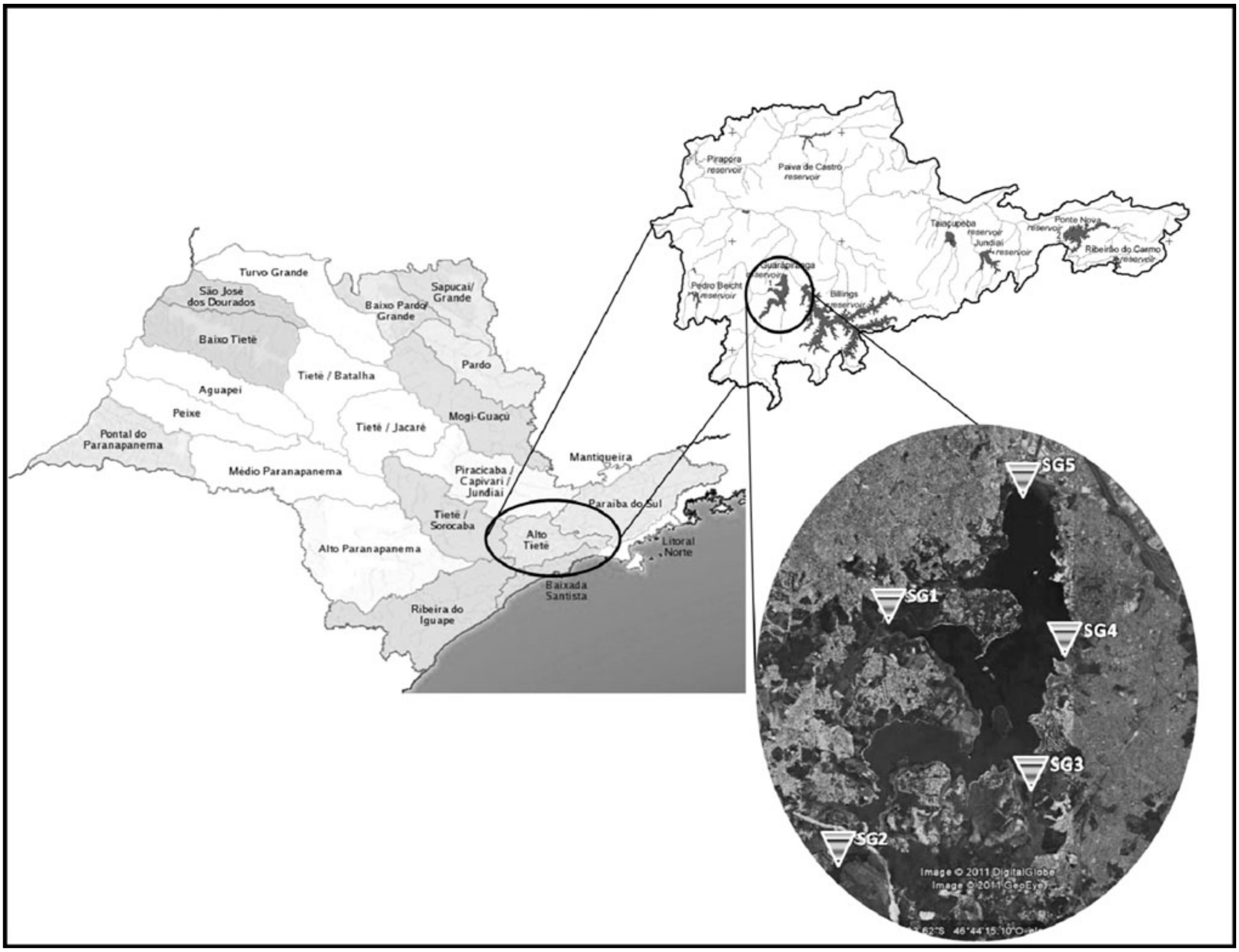

Fig. 1 Guarapiranga Reservoir and location of the sampling points

Table 1 Sampling points location in the Guarapiranga Reservoir

Samplings points	Location	Geographical position (GPS)	Depth (m)
SG01	Tributary of the Embu-Mirim river, about 300 m downstream the Cumbica highway	S 23°42′37.50″ W 046°46′026″	3.5
SG02	Tributary of Embu-Guaçú river, about 1 km upstream Rodoanel bridge	S 23°46′153″ W 46°46′564″	6.0
SG03	Tributary of the Parelheiros river, about 1 km upstream Ilha dos Eucaliptos	S 23°45′107″ W 46°43′318″	4.1
SG04	In the middle of the reservoir, in front of the Castelo country club	S 23°43′021″ W 46°43′213″	8.5
SG05	In the middle of the reservoir, in front of Guarapiranga Park, about 500 m upstream from SABESP capitation point	S 23°40′351″ W 46°43′434″	

Granulometric analysis and organic matter content of the sediment samples

These chemical analyses were carried out at the Instituto Agronômico de Campinas (IAC). The granulometric analysis was done according to Camargo et al. [10] by using pipetting methodology.

Multielemental determination by INAA

Samples and reference materials were sealed in pre-cleaned double polyethylene bags for irradiation. Single and multi-element synthetic standards were prepared by pipetting convenient aliquots of standard solutions (SPEX CERTIPREP) onto small sheets of Whatman No. 41 filter paper. In the IEA-R1 nuclear reactor at IPEN the sediment samples, reference materials and synthetic standards were irradiated for 8 s (short irradiation) and 8 h (long irradiation) and counted at different times depending on the radionuclide half-live produced in the irradiation, under a thermal neutron flux of 1–5 × 10^{12} n cm^{-2} s^{-1}. Gamma spectrometry was performed using a Canberra gamma X hyperpure Ge detector and associated electronics, with a resolution of 0.88 keV and 1.90 keV for ^{57}Co and ^{60}Co, respectively. The elements As, Ba, Br, Co, Cr, Cs, Cu, Fe, Hf, K, Mn, Na, Rb, Sb, Sc, Ta, Th, Ti, U, V, Zn and the rare earth elements Ce, Eu, La, Lu, Nd, Sm, Tb and Yb were determined during long irradiation. The elements Mn, Ti and V were determined by short irradiation. The analysis of the data was done by using in-house gamma ray software, VISPECT program to identify the gamma-ray peaks and by an ESPECTRO program to calculate the concentrations. Detailed analytical methodology is described at Larizzatti et al. [11]. The precision and accuracy of both methodologies were verified by using the Z score according to Bode [12] for Buffalo River Sediment, Soil 7 and BEN-Basalt reference materials analyses.

Results and discussion

The mean values (mg kg^{-1}) for the duplicate and standard deviation for the elements analyzed by INAA for the sediment samples collected in the April of 2009 and June of 2010 are shown in Table 2. This table also lists the values for the Upper Continental Crust, UCC [13], and the concentrations obtained for the mean values in a set measurement made in the local soil [14]. The observed concentration in the regional soil considerably differs from the UCC values for several of the analyzed elements. The concentration observed in sediment samples resembles much more the soil pattern than that for UCC (Fig. 2a) or even than the North American Shale Composite, NASC [15], another geochemical reference widely used in sediment studies. It is also worthwhile to note that geochemical differences separate points SG1 from SG2 and SG3 and these from SG4 and SG5.

To assess the elemental pattern distribution in an environmental system multivariate analysis is frequently used [16, 17]. Pearson's correlation coefficients among the content of the elements indicated significant positive correlations between (a) Fe, Mn, Ti, V, Sc, As, Ca, Br, Sb and Na ($r > 0.63$), (b) Th, Cr, Zn ($r > 0.65$), (c) light rare earth elements LREE—La, Ce, Nd, Sm and Eu ($r > 0.70$), (d) K, Co, Cs, Hf, Lu, Tb and Yb ($r > 0.58$). Significant negative correlations were found for LREE and (b) and for (d) ($r < -0.45$). The elements from (a) and (b) were found to be weakly correlated (r from 0.30 to 0.59). The organic matter (OM) content showed good agreement with the clay content ($r = 0.73$) and both showed significant positive correlation only with LREE (r from 0.76 to 0.97). Silt content only showed good correlations to (b) (r from 0.78 to 0.81) and the clay content presented significant positive correlations with Hf, K and HREE—heavy rare earth elements, Tb, Yb and Lu (r from 0.64 to 0.69).

Table 2 Results for the elements analyzed by INAA (mg kg^{-1}): mean, standard deviation, soil [14] and UCC values [13]

	SG1-1	SG1-2	SG2-1	SG2-2	SG3-1	SG3-2	SG4-1	SG4-2	SG5-1	SG5-2	Soil	UCC
As	5.3 ± 0.4	3.3 ± 0.2	10.5 ± 1.0	9.2 ± 0.4	8.3 ± 0.2	9.8 ± 0.4	14.7 ± 1.1	9.0 ± 0.3	12.7 ± 0.8	10.7 ± 0.4	5.7	2
Ba	467 ± 30	435 ± 34	680 ± 43	365 ± 22	321 ± 22	619 ± 42	209 ± 14	374 ± 24	344 ± 29	454 ± 60	219	668
Br	12.3 ± 0.2	14.3 ± 0.3	12.5 ± 0.1	10.9 ± 0.3	10.0 ± 0.1	14.6 ± 0.3	17.1 ± 0.2	21.4 ± 0.3	23.5 ± 0.3	28.3 ± 0.4	n.d.	1.6
Ca (%)	1.34 ± 0.04	1.24 ± 0.04	1.51 ± 0.03	1.18 ± 0.06	1.61 ± 0.04	0.90 ± 0.06	2.45 ± 0.06	1.76 ± 0.05	1.73 ± 0.03	1.61 ± 0.07	0.05	2.94
Co	9.5 ± 0.2	7.4 ± 0.1	11.4 ± 0.2	10.8 ± 0.1	8.5 ± 0.1	9.0 ± 0.2	9.1 ± 0.2	8.3 ± 0.1	9.4 ± 0.2	9.7 ± 0.1	6.7	11.6
Cr	49 ± 2	47 ± 1	78 ± 4	74 ± 2	75 ± 3	77 ± 2	80 ± 3	84 ± 2	80 ± 4	78 ± 2	63.0	35
Cs	6.2 ± 0.2	5.5 ± 0.5	8.3 ± 0.3	8.1 ± 0.7	10.1 ± 0.4	10.0 ± 1.9	6.7 ± 0.3	6.6 ± 0.7	6.7 ± 0.3	6.5 ± 0.8	2.86	5.8
Fe (%)	4.82 ± 0.03	3.99 ± 0.03	4.56 ± 0.03	4.18 ± 0.02	3.51 ± 0.11	4.27 ± 0.03	6.13 ± 0.20	6.25 ± 0.04	6.04 ± 0.20	5.71 ± 0.04	2.90	3.09
Hf	2.4 ± 0.1	2.3 ± 0.1	13.9 ± 0.3	13.8 ± 0.6	10.4 ± 0.2	6.9 ± 0.3	7.4 ± 0.1	7.5 ± 0.3	6.7 ± 0.1	6.2 ± 0.2	9.8	5.8
K (%)	0.80 ± 0.13	0.71 ± 0.02	2.48 ± 1.14	n.d.	1.42 ± 0.3	1.28 ± 0.08	0.81 ± 0.23	0.84 ± 0.24	0.73 ± 0.15	n.d.	0.86	2.86
Mn	301 ± 19	221 ± 8	460 ± 45	743 ± 29	320 ± 25	202 ± 2	946 ± 83	989 ± 35	1124 ± 32	1274 ± 39	392	527
Na (%)	0.085 ± 0.011	0.083 ± 0.006	0.100 ± 0.003	0.107 ± 0.001	0.094 ± 0.005	0.082 ± 0.004	0.116 ± 0.012	0.134 ± 0.005	0.063 ± 0.004	0.046 ± 0.011	0.09	2.57
Rb	139 ± 8	44 ± 3	92 ± 4	85 ± 5	93 ± 4	84 ± 11	42 ± 2	55 ± 3	68 ± 4	43 ± 3	38	110
Sb	1.34 ± 0.06	1.26 ± 0.18	0.80 ± 0.04	0.75 ± 0.11	1.22 ± 0.08	0.94 ± 0.08	1.58 ± 0.07	1.57 ± 0.12	1.46 ± 0.11	1.14 ± 0.06	0.85	0.31
Sc	15.1 ± 0.2	14.9 ± 0.2	17.2 ± 0.2	16.2 ± 0.3	14.5 ± 0.2	15.6 ± 0.3	17.1 ± 0.2	17.2 ± 0.3	15.7 ± 0.2	15.3 ± 0.3	9.2	7
Sm	9.6 ± 0.1	9.20 ± 0.09	7.3 ± 0.1	7.06 ± 0.07	7.40 ± 0.10	7.43 ± 0.07	6.8 ± 0.1	6.65 ± 0.06	7.20 ± 0.1	6.89 ± 0.07	6.78	4.7
Ta	2.3 ± 0.2	2.0 ± 0.3	2.6 ± 0.2	2.1 ± 0.2	2.1 ± 0.2	3.2 ± 0.2	1.8 ± 0.1	2.6 ± 0.1	2.5 ± 0.2	2.7 ± 0.1	1.22	1.5
Th	17.8 ± 0.5	17.4 ± 0.3	18.6 ± 0.5	16.9 ± 0.3	22.0 ± 0.6	22.5 ± 0.5	24.4 ± 0.7	27.7 ± 0.5	21.7 ± 0.6	20.2 ± 0.4	18.0	10.3
Ti (%)	0.58 ± 0.06	0.52 ± 0.05	0.79 ± 0.04	0.63 ± 0.07	0.58 ± 0.06	0.48 ± 0.04	0.94 ± 0.15	0.64 ± 0.02	0.78 ± 0.03	0.58 ± 0.04	0.58	0.31
U	8.4 ± 0.4	8.7 ± 0.3	5.2 ± 0.2	5.1 ± 0.2	8.5 ± 0.4	9.9 ± 0.3	7.5 ± 0.4	7.3 ± 0.2	7.5 ± 0.3	7.1 ± 0.2	5.4	2.5
V	101 ± 10	91 ± 15	115 ± 8	90 ± 14	83 ± 8	72 ± 4	150 ± 10	128 ± 7	126 ± 6	87 ± 5	78	53
Zn	129 ± 4	81 ± 3	152 ± 5	152 ± 4	193 ± 6	200 ± 12	209 ± 7	231 ± 9	169 ± 5	140 ± 5	48	52

n.d. Not determined

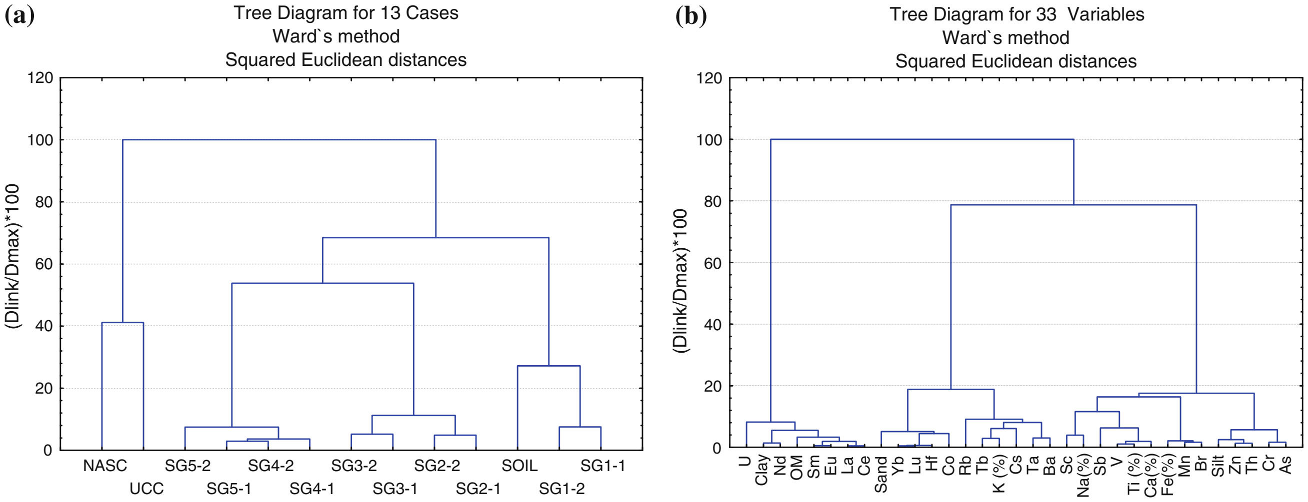

Fig. 2 Dendrogram in **a** Q-mode, cases, and in **b** R-mode, variables, for the samples of Guarapiranga Reservoir

Table 3 Results for granulometric analysis and OM content for the sediment samples

Sample	Organic matter content (g dm^{-3})	Clay (%)	Silt (%)	Total sand (%) (2.00–0.053 mm)	Textural classification
SG1-1	109.0	65.4	25.8	8.8	Very clayish
SG1-2	n.d.	68.1	22.7	8.2	Very clayish
SG2-1	59.3	38.9	43.1	18.0	Mostly clay silt
SG2-2	42.0	28.1	24.9	47.0	Mostly clay sand
SG3-1	56.0	41.4	48.6	10.0	Clay silt
SG3-2	51.0	54.7	47.4	6.9	Clay silt
SG4-1	59.0	48.7	50.2	1.1	Clay silt
SG4-2	56.0	51.0	46.8	2.2	Clay silt
SG5-1	68.0	56.5	41.9	1.6	Clay silt
SG5-2	60.0	53.6	44.0	2.4	Clay silt

SGX-1 first campaign, *SGX-2* second campaign, *n.d.* not determined

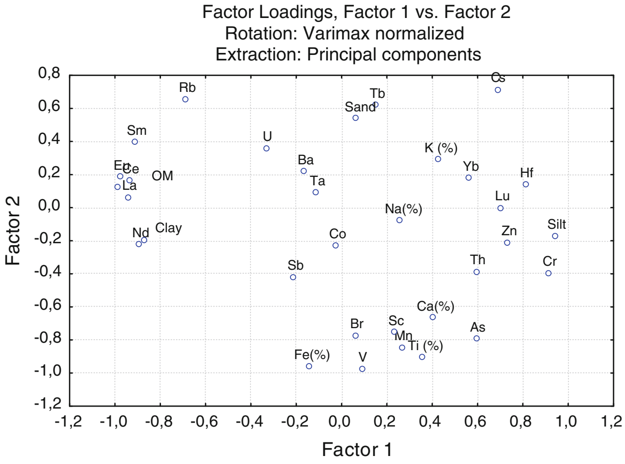

Fig. 3 Plot of factors 1 and 2 extracted from the principal component factor analysis

The results for the granulometric analysis and OM content for the sediment samples are presented in Table 3. Points SG1 and SG5 presented the highest OM content followed closely by points SG2, SG3 and SG4. The same pattern can be observed for the REE distribution with higher concentrations of LREE in points SG1 and SG5 and higher concentrations of HREE in points SG2, SG3 and SG4. Regarding granulometric distribution SG1 presented the highest clay and lowest silt percentage and this point was the one with lower concentrations of As, Cr, Hf, Mn, Th and Zn. Points SG3, 4 and 5 presented a very similar granulometric composition, very similar clay and silt proportions. SG2-1 (point SG2, first campaign), presented a higher silt percentage than clay in its composition and SG2-2, presented higher clay percentage than silty. This point presented the highest sandy fraction, 18 and 47%, respectively and also the highest Hf concentration reflecting the Hf and sand correlation probably due to the presence of hafnium bearing minerals [18].

Figure 2b shows the dendrogram obtained in the Cluster Analysis for the determined elements. Two main groups were formed. A first group composed bay clay, OM and the associated elements (U and LREE). The second group was

Table 4 Total concentration (mg kg^{-1}) of ∑REE, ∑LREE, ∑HREE, and ratios of, La/Sm, La/Yb, Ce/La, Eu/Sm, Yb/Sm, and Ce/Ce* in sediments and soil from the Guarapiranga Reservoir [14], and NASC values [13]

	SG1-1	SG1-2	SG2-1	SG2-2	SG3-1	SG3-2	SG4-1	SG4-2	SG5-1	SG5-2	Soil [15]	NASC [14]
La	62.70	61.75	40.90	39.91	43.70	46.10	48.60	47.92	44.50	43.73	50.4	32.0
Ce	134.2	125.5	85.90	80.01	96.90	91.03	98.50	96.41	100.8	81.79	130.4	73.0
Nd	48.90	51.70	33.00	30.50	33.70	35.40	39.10	34.90	42.70	47.50	47.0	33.0
Sm	9.60	9.20	7.30	7.06	7.40	7.43	6.80	6.65	7.20	6.89	8.05	5.7
Eu	2.16	1.95	1.28	1.09	1.26	1.27	1.25	1.11	1.41	1.39	1.09	1.24
Tb	0.80	0.79	1.00	0.87	0.90	n.d.	0.55	0.77	0.80	0.94	1.27	0.85
Yb	2.00	1.64	5.30	4.94	3.43	3.66	2.50	2.82	2.60	2.57	4.24	3.1
Lu	0.30	0.27	0.83	0.82	0.57	0.59	0.55	0.56	0.42	0.39	0.54	0.48
∑REE	260.7	252.8	175.5	165.2	187.9	185.5	197.9	191.1	200.4	185.2	243.0	149
∑LREE	257.6	250.1	168.4	158.6	183.0	181.2	194.3	187.0	196.6	181.3	236.9	145
∑HREE	3.1	2.7	7.1	6.6	4.9	4.3	3.6	4.1	3.8	3.9	6.0	4
∑LREE/∑HREE	83.1	92.6	23.6	23.9	37.3	42.6	54.0	45.1	51.5	46.4	39.2	33
La/Yb	31.4	37.7	7.7	8.1	12.7	12.6	19.4	17.0	17.1	17.0	11.9	10.3
La/Sm	6.5	6.7	5.6	5.7	5.9	6.2	7.1	7.2	6.2	6.3	6.3	5.6
Ce/La	2.1	2.0	2.1	2.0	2.2	2.0	2.0	2.0	2.3	1.9	2.6	2.3
Eu/Sm	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.1	0.2
Yb/Sm	0.2	0.2	0.7	0.7	0.5	0.5	0.4	0.4	0.4	0.4	0.5	0.5
Ce/Ce*	1.01	0.97	0.92	0.88	1.00	0.91	1.00	0.99	1.04	0.87	1.20	1.0

n.d not determined Ce/Ce* = 2(Ce/Ce$_{\text{shale}}$)/(La/La$_{\text{shale}}$ + Sm/Sm$_{\text{shale}}$)

also divided into two sub-groups: one comprising the silt portion and the main metallic elements (Ca, Cr, Fe, Mn, Sc, Th, Ti, V and Zn) and semi-metals As and Sb. The other sub-group formed by sand in association with HREE, Ba, Co, Cs, Hf, K, Rb and Ta. This result indicates that the distribution of the elements is mainly controlled by the different phases of gramulometric content and that the OM is closely correlated with clay content.

Similar results were obtained from the application of Factor Analysis (Fig. 3). Four factors could be extracted and accounts for 100% of the observed variance. Factor 1 explains 42.39% of the total variance; Factor 2 is responsible for 35.52%; Factor 3, for 14.62% and Factor 4, 7.47% of the total variance. Factor F1 comprises (the −signal are being used to indicate a negative loading value inside the factor): -Ce, Cr, -Eu, Hf, -La, Lu, -Nd, -Sm, Zn, -OM and -clay; F2 comprises: -As, -Br, Cs, -Fe, -Mn, -Sc, -Ti and -V; F3 comprises: Ba, Co, K, -Sb, Ta, -U, Yb and sand and the F4 comprises: -Na. The inverse correlation between the LREE and Cr and Zn suggest different origin for these elements and also direct correlation between Fe, Mn, Ti and V indicates a same source for these elements to be incorporated in the sediment.

The rare earth elements (REE) concentrations are presented in Table 4 together with the total content of REEs (∑REE), light REEs (∑LREE), heavy REEs (∑HREE) and REE ratios. The (∑REE) shows that the points SG2, 3, 4 and 5 are depleted in relation to the soil content and an enrichment of LREE, evidenced by the ratios ∑LREE/∑HREE and La/Yb, can be observed in points SG1, 4 and 5. No fractionation occurs among the LREE as evidenced by the ratios La/Sm and La/Ce as well as no Ce anomaly could be observed. These results indicated that the geochemical factors that caused the LREE/HREE fractionation did not affect the LREE distribution and the redox conditions during the REE mobilization was not oxidizing dependent [19]. These results also corroborate the fact that the points of collection have different source of origin, one rich in LREE, points SG1, 4 and 5, and the other with REE content similar to regional soil and NASC content and distribution pattern.

The crustal origin of sediments and depositional processes can be traced by the mean of elemental ratios [20] and lithophile elements are generally used with this in mind. In the regional soil the ratios Th/Sc and Cr/Sc presented values of 1.6 and 4.9, respectively. In the sediment samples these ratios varied in a narrow range of 1.0 to 1.6 and 3.1 to 5.2, respectively. Also the ratios La/Th and Co/Th, that in soil presented the values 2.5 and 0.32, respectively showed little variation in sediment samples: 1.7 to 3.6 and 0.30 to 0.64, respectively. The results indicate detrital origin for these elements. On the other hand, comparing the results of sediment with the upper continental crust and the same values for shale (NASC) it is possible to conclude that the region has high background levels for Th and U. This was reveled by the following ratios: Co/Th = 1.13 and 2.33; Th/U = 4.1 and 4.4 and U/Sc = 0.36 and 0.18 in UCC and NASC respectively, while in the sediment samples the observed values for these ratios were: 0.30 to 0.64; 2.0 to 3.8 and 0.30 to 0.63 respectively.

Conclusions

Sediments from five points, collected in two sampling campaigns in the Guarapiranga Reservoir were analyzed by instrumental neutron activation analysis. The results were compared with reference soil of the region, UCC and NASC in order to determine the geochemical factor that controls the distribution of the trace elements in these sediments. It was possible to verify that, at least, two different sources contribute for the sediment processes in the reservoir: one that affects the points SG1, 4 and 5 and one distinct source that contributes for points SG3 and 4 sedimentary processes. In the first case the sediments are affected by processes that lead to a fractionation of the LREE related to the HREE. The mineralogical settings correlated with the granulometric distribution poses the following characteristics for the elements distribution: LREE distribution is controlled by the clay content, transition metals are mainly controlled by the silt content and HREE, Hf, K and Co are linked to the sand content. The study of elemental ratios indicated that the amount of elements present in the Guarapiranga Reservoir is mainly of detrital origin.

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