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Determination of Cr, Cu, Ni, Pb and Zn in the sediments of Mangueira Lagoon/RS/Brazil

The Mangueira Lagoon is 123 km long and 7.6 km wide at its widest point, with an average depth of 2.5 m. By the fact that this lagoon is one of the youngest geological formations on Earth (only 4.5 thousand years), little is known about the geology of its sediments. This work describes the concentration of the toxic metals Cr, Cu, Ni, Pb and Zn in surface sediments samples of Mangueira Lagoon. The sediment samples were collected and underwent partial acid digestion (aqua regia / perchloric acid). The extracts obtained were analyzed by atomic absorption spectroscopy in flame. Cu and Zn were detected at all points during the test. Zn is a major element, ranging from 2.5 mg kg-1 ± 0.3% to 4.1 mg kg-1 ± 2.4%, followed by Cu with values between 2.0 mg kg-1 ± 8.5% to 2.5 mg kg-1 ± 4.2%. Considering the parameters established by the CCME (Canadian Environmental Quality Guidelines), TEL (Threshold Effect Level) and PEL (Probable Effect Level), the concentrations of toxic metals found in the surface sediment were low and showed low environmental impacts. These results constitute a contribution to a better understanding of the processes that occur in the region, serving as a basis for future work.

Keywords: Atomic Absorption; Environmental Characterization; Toxic Metals.

Determinação de metais tóxicos Cr, Cu, Ni , Pb e Zn em amostras de sedimentos da Lagoa Mangueira/RS/Brasil

A Lagoa Mangueira tem 123 km de extensão por 7,6 km de largura no seu ponto mais largo e uma profundidade média de 2,5 m é uma das formações geológicas mais jovens da Terra, com apenas 4,5 mil anos, pouco se conhece sobre a geologia de seus sedimentos. Este trabalho descreve a concentração dos metais tóxicos Cr, Cu, Ni , Pb e Zn em amostras de sedimentos superficial da Lagoa Mangueira. As amostras de sedimentos foram coletadas e sofreram digestão pseudo-total (água régia/ ácido perclorico), sendo os extratos analisados por espectroscopia de absorção atômica em chamas. De forma geral todos os metais foram detectados. Sendo apenas Cu e Zn detectados em todos os pontos. O Zn apresenta-se como elemento majoritário variando de 2,5 mg kg-1 ± 0,3% a 4,1 mg kg-1 ± 2,4%, seguido pelo Cu com valores entre 2,0 mg kg-1 ± 8,5% a 2,5 mg kg-1 ± 4,2%. Considerando os parâmetros estabelecidos pelo CCME (Canadian Environmental Quality Guidelines) TEL (Threshold Effect Level) e PEL (Probable Effect Level), as concentrações de metais tóxicos encontradas no sedimento superficial foram baixas e demonstraram baixo impacto ambiental. Estes resultados constituem-se numa contribuição para o melhor entendimento dos processos que ocorrem na região, servindo de base para futuros trabalhos.

Palavras-chave: Absorção Atômica; Caracterização Ambiental; Metais Tóxicos.

Topic: **Desenvolvimento, Sustentabilidade e Meio Ambiente**

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Marcelo Uarthe Grimmler
Instituto Federal de Educação, Ciência e Tecnologia Sul-Rio-Grandense, Brasil
http://lattes.cnpq.br/5722981894689116
marcelogrimmler@hotmail.com

Francisco Osvaldo Peres Pereira
Instituto Federal de Educação, Ciência e Tecnologia Sul-Rio-Grandense, Brasil
http://lattes.cnpq.br/3744526671789412
frangian@terra.com.br

Lucas Aldrigui Silveira
Instituto Federal de Educação, Ciência e Tecnologia Sul-Rio-Grandense, Brasil
http://lattes.cnpq.br/5225792036428553
lucas.aldrigui@gmail.com

Leandro dos Santos
Instituto Federal de Educação, Ciência e Tecnologia Sul-Rio-Grandense, Brasil
http://lattes.cnpq.br/0888460193720312
leokimico@hotmail.com

Pedro José Sanches Filho
Instituto Federal de Educação, Ciência e Tecnologia Sul-Rio-Grandense, Brasil
http://lattes.cnpq.br/9785390634457316
http://orcid.org/0000-0002-9852-8940
pjsans@ibest.com.br

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INTRODUCTION

The state of Rio Grande do Sul is privileged with its coastal plain and abundance of rivers, lakes and lagoons with great water volumes, especially water environments such as Patos' lagoon of, Mirim lagoon and Mangueira lagoon (SCHAFER, 2009). The Mangueira lagoon is 123 km long and 7.6 km wide at its widest point and an average depth of 2.5 m, presenting neither tributaries nor superficial communication with the Atlantic Ocean, resulting in a total area of 800 km^2 (VIEIRA et al., 1988). It is located between the municipalities of Rio Grande, Santa Vitória do Palmar and Chuí, with a distance of more than 500 km from the Porto Alegre (State's Capital City). Is situated almost on the border with Uruguay, with no urban concentrations nearby. It is one of the youngest geological formations on Earth, being only 4.5 thousand years old (TOMAZELLI et al., 2005). Its surrounding area has preferential uses linked to agriculture, livestock, fishing and recreation. The zone located between this lagoon and the sea is dominated by permeable sediments, forming a zone of transition between the superficial water and groundwater (ULLMAN et al., 2003).

Once a sea, Mangueira lagoon is unique: an immense freshwater on a bed full of fossil shells, populated by fish like Hoplias malabaricus and Atherina presbyter and by mammals like capybara. Because of its very high pH, due to the soil composition, the Mangueira lagoon is home to a microalga that is beneficial to human health and capable of absorbing large amounts of pollutants from the atmosphere (POUEY and BRITTO, 2012). While physical diversity and biological productivity are characteristic of these systems, their fragility is also recognized in the face of anthropogenic aggression (SCHAFER, 2009).

A few studies were carried out in Mangueira Lagoon for analysis of kingfish (Atherina presbyter) feeding (PIEDRAS, 2005); development of a hydrodynamic model of nutrients and plankton transport for shallow bodies by Fragoso et al (2009); dominance and structure of fish size by Artioli et al. (2009), among others. However, there is nothing related to the physical-chemical characterization of the sediment and its levels of toxic metals.

Sediments play a key role in the bioavailability of various chemical compounds and can be used to detect the presence of contaminants, as it is a compartment of great importance in the evaluation of contamination and pollution of aquatic ecosystems. In low-flow sites, precipitation and deposition of sediments are facilitated, which reflects that it has the same chemical characteristics of a water column, as it incorporates pollutants through various processes such as adsorption and complexation with organic matter (JESUS et al., 2004). By accumulating different contaminants, the sediment becomes a good indicator for monitoring the aquatic ecosystem (ARRIBÉRE et al., 2003). On the other hand, changes in the physical-chemical conditions of this reservoir can reprocess these materials making them available again in the middle parts (MARIANI et al., 2011).

The study of toxic metals in aquatic environments in Rio Grande do Sul has been the focus of several studies, such as Betemps et al. (2012) - developed a seasonal study of toxic metals in the sediment of the 'Saco do Laranjal' in Patos' Lagoon; Santos et al. (2003) – developed study of toxic metals in Mirim Lagoon sediments, among others.

The evaluation of the levels of toxic metals in the Mangueira Lagoon is of extreme importance in order to evaluate changes in this delicate ecosystem, since its waters are used to irrigate the region's agriculture and for artisanal fisheries. The waters used in agriculture leach the crops, returning to the lagoon carrying fertilizers, agricultural pesticides, used in these crops (ANDRADE et al., 2008). Due to lack of data on levels of toxic metals in Mangueira Lagoon, the objective of this work is to determine the concentration of the toxic metals Cr, Cu, Ni, Pb and Zn in the surface sediments of this lagoon and to serve as a basis for future studies in this environment.

MATERIALS AND METHODS

The Mangueira lagoon is located in the southern region of the State of Rio Grande do Sul, between the dunes that separate the municipality of Santa Vitória do Palmar and the Atlantic Ocean, with geographical coordinates (33 ° 05'27.02 '' S and 52° 46' 03.01" W). For the development of this study, five sediment samples were collected on a stainless-steel dredger of the Van Veen type, as indicated by Mozeto (2007), in October 2015. Point one (P1) was collected where a drainage channel of irrigation water leached from rice fields (33°1'49 "S and 52°42'23" W), point two (P2) (32°58'53"S and 52°40'9" W) and point three (P3) (32°55'34 "S and 52°38'42" W) north of the lagoon, point four (P4) (33°4'18 "S and 52°45'33" W) and point five (P5) (33°8'3 "S and 52°46'8" O) further south. Figure 1 shows the location of collection points.

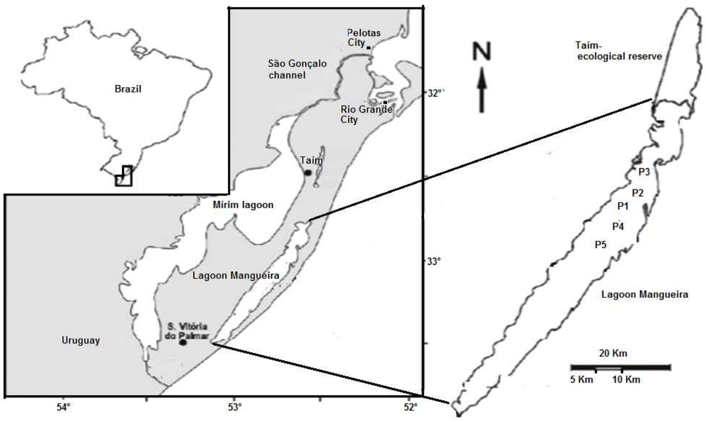

Figure 1: Location of Mangueira lagoon and collection points, adapted from Artioli (2009).

The exact position of each sampling point (Figure 1) was defined by GPS measurements (GPS, Global Positioning System, Garmin & ETrex® equipment, accuracy of ± 15 m). Samples were collected and stored in previously decontaminated polyethylene pots. After collection, the samples were duly transported and stored at 4°C. Part of the samples was reserved for determination of moisture, organic matter (O.M.), particle size and pH.

The determination of the organic matter in the sediment occurred by volatile losses, at a temperature of 550°C, for 4 hours (APHA, 2005). Grain (2 mm), very coarse sand (1 mm), coarse sand (500 µm), medium sand (250 µm) and fine sand (125 µm), measured using the Wertworth scale, very fine sand (63 µm), bottom (<63 µm). This method was described by Suguio (1973). The moisture was determined with 10 g of sample in an oven at 105°C for 24 hours (APHA, 2005). The pH of the sediment was determined in suspension using the ratio 1: 2 (sediment / water from the same point) as employed by Yoon et al. (2006).

For the extraction of metals, partial acid digestion was used, which evaluates the level of metals in the sediment that may become bioavailable (HORTELLANI et al., 2005). The collected samples were dried in an oven at 60 °C for 48 hours and soon after macerated and sifted, being used in the fraction of <63 µm. In 2 g of this fraction (in triplicate) were added 4 mL of "aqua regia" (3: 1, HCl: HNO3), 4 mL of distilled water and 1 mL of perchloric acid ($HClO_4$). The solution was heated in water bath for 30 minutes at a temperature of 90°C, procedure adapted from Hortellani et al. (2005).

The solution was filtered and transferred to a volumetric flask of 25.0 mL and its volume was corrected with ultra-pure water. For analytical certification, a sample of reference material NMCR #4 (Natural Matrix Certified Reference) for soil and sediment obtained from Ultra Scientific Analytical Solutions was analyzed in parallel. Limits of detection (LoD) and quantification (LoQ) were determined by ten measurements of blanks in the atomic absorption spectrophotometer. The LoD was considered as the average of analytical signal of the blank plus three times the standard deviation; for the LoQ, was carried out in the same way, but using 10 times the standard deviation of the blank (IUPAC, 1997). The determination of the metals (Cr, Cu, Ni, Pb and Zn) was performed by PerkinElmer - Analyst 200 Flame Atomic Absorption Spectrophotometry. For quantification of the metals, calibration curves were constructed between 0.2 and 4.0 mg L^{-1} by dilutions of Titrisol®-Merck 1000 mg L^{-1} standard solutions, undergoing the same treatment of the samples. The operating conditions in the spectrophotometer for the metals analyzed are presented in table 1.

Table 1: Operating conditions of the Atomic Absorption spectrophotometer.

metal	Wave-length (nm)	Lamp current value (mA)	Flame type
Pb	283.3	10.0	Air-acetylene
Cu	324.7	15.0	Air-acetylene
Zn	213.9	15.0	Air-acetylene
Cr	357.8	25.0	Nitrous oxide-acetylene
Ni	232.0	25.0	Air-acetylene

nm: nanometer; mA: miliamper.

The material used in the treatment and storage of samples, such as glassware in general, was decontaminated with a solution of 10% (v/v) HNO_3 for 24 hours, with subsequent oven drying at 105°C.

RESULTS AND DISCUSSION

Table 2 shows the results obtained in the sediment analysis for organic matter, moisture and pH. We can verify that the average value of pH is 7.04, with its maximum (7.17) and minimum (6.91). It is shown that the Mangueira lagoon sediments has a neutral pH. The data suggests that the points near the entrance (of the lagoon), where water leaches through the plantations, leads to a reduction of pH, which gradually rises as we advance inside the Lagoon. The organic matter contents were low, evidencing the low deposition of matter of vegetable or animal origin, being characterized as a sediment of mineral origin.

Table 2: Preliminary characterization of the sediment - Moisture content- (% ± RSD%), Organic matter content-OM (% ± RSD%) and pH.

Points	Moisture (%)	OM (%)	pH
P1	25.1±2.1	0. 55±3.5	6.91
P2	25.2±1.9	0.44±3.3	6.92
P3	26.1±1.8	0.43±3.4	7.10
P4	29.1±2.2	0.70±3.1	7.13
P5	23.0±2.0	0.36±3.6	7.17

The granulometric distribution presented in table 3 shows the predominance of the fine sand fraction (125 μm) in the sediment. These data, together with OM levels, suggest unfavorable conditions for the deposition of heavy metals in this environment. The values of moisture varying between 29.1% and 23.0% may be related to interstitial water and agree with the low content of fines.

Table 3: Granulometric Distribution (%).

	Grain	Very coarse sand	Coarse sand	Average sand	Thin sand	Very fine sand	Bottom < 63µm
P1	0	0.15	1.05	8.38	84.28	5.84	0.29
P2	0.02	0.15	0.72	7.21	85.7	6.01	0.20
P3	0	0.13	1.01	7.37	84.86	6.45	0.17
P4	0.19	1.07	2.4	12.66	78.99	4.59	0.10
P5	0.06	0.76	0.22	6.28	86.7	5.77	0.20

Table 4 presents the main characteristics of the analytical method used. It can be observed that the analysis by atomic absorption spectrophotometry was linear for all analytes, with linear correlation coefficients ranging from 0.999 to 0.997. The angular coefficient values (a) suggest that the determination of zinc levels occurred with greater sensitivity. The recoveries for the certified material was above 90%, with the exception of chromium. According to Jesus et al. (2004), recoveries between 75 and 110% for toxic metals are considered acceptable. The lower recovery values presented for chromium can be explained by the fact that it is associated with the silicates in the reference material that are not extracted by the digestion method used in this study (HORTELLANI et al., 2005).

Table 4: Figure of Merit; Limits of detection and quantification in mg kg-1 and mg L^{-1}, angular and linear coefficients of the calibration curves.

	a	b	R^2	RSD(%)	CV	FV	%Rec.	LD (mg kg^{-1})	LQ (mg kg^{-1})	LD (mg L^{-1})	LQ (mg L^{-1})
Cu	0,0433	0,0033	0,999	1,5	36,4	35,2	96,7	0,1	0,2	0,01	0,02
Ni	0,0473	-0,0005	0,997	0,9	26,0	23,5	90,4	0,2	0,6	0,02	0,05
Pb	0,0133	-0,0017	0,999	1,2	95,3	89,9	94,3	0,6	2,1	0,05	0,17
Cr	0,0127	0,0142	0,988	2,1	48,1	31,0	64,4	0,3	1,1	0,02	0,09
Zn	0,1101	-0,0422	0,998	1,2	133,5	128,3	96,1	0,1	0,4	0,01	0,03

LD: Detection Limit; LQ: Quantification Limit; a: angular coefficient; b: linear coefficient; CV: certified value; FV: found value.

Table 5 presents the results in mg kg^{-1} of the toxic metals analyzed. In general, all metals were detected. However, Cu and Zn were measured at all points. Zn is the major element, ranging from 2.5 mg kg^{-1} ± 0.3% to 4.1 mg kg^{-1} ± 2.4%, followed by Cu with values between 2.0 mg kg^{-1} ± 8.5% to 2.5 mg kg^{-1} ± 4.2%.

According to Silva (2009), the presence of Zn is related to the geology of the region. This situation was also observed by Betemps and Sanches (2012), in their study in "Saco do Laranjal”. Copper presence may be related to agricultural activities. Rosa (2002) describes in her work that copper levels determined in the central part of the Patos’ Lagoon could have been carried by the waters of the Camaquã River, which captures water from plantations, where many agricultural defenses are composed of copper.

Table 5: Metal levels in mg kg-1 (± RSD) found in Mangueira lagoon sediments, and PEL and TEL levels.

	P1	P2	P3	P4	P5	TEL(1)	PEL(2)
Cu	2.1±10.1	2.1±5.5	2.0±8.5	2.4±11.2	2.5±4.2	35.7	197.0
Cr	D	D	D	D	D	37.3	90.0
Ni	ND	ND	ND	ND	0.7±6.2%	18.0	35.9
Pb	ND	ND	D	ND	ND	35.0	91.3
Zn	4.0±3.6%	3.2±3.8%	2.5±0.3%	2.5±1.3%	4.1±2.4	123.0	315.0

(1) TEL: Threshold Effect Level. Value below which biological effects rarely occur (Freshwater); (2) PEL: Probable Effect Level. Value above which adverse effect is expected (Fresh water); D: detected; ND: Not detected.

The values of toxic metals quantified at all points did not exceed the limits of TEL (Threshold Effect Level: value below which harmful effects to biological systems rarely occurs) and PEL (Probable Effect Level: value above which adverse effects occur to biological systems) (CCME, 2002). The values given for TEL and PEL correspond to the level 1 and level 2 values established in CONAMA Resolution 344/2004 for the quality of the water sediments (CONAMA, 2004).

The values of toxic metals studied in Lagoa Mangueira were lower than those found in the Mirim and Patos' lagoons. In the Mirim Lagoon, 12.7 mg kg^{-1} of Cu and 55.9 mg kg^{-1} of Zn were quantified in the sediment (Santos, 2003) and in 'Saco do Laranjal' (in Lagoa dos Patos) 6.7 mg kg^{-1} of Cu and 20.6 mg kg^{-1} of Zn were quantified, demonstrating a low level of contamination by these metals in Mangueira Lagoon.

CONCLUSIONS

The concentrations of toxic metals found in the surface sediment showed low environmental impact, due to the reduced urbanization around them and restricted anthropogenic action in their environment (agricultural activity). These results constitute a contribution to a better understanding of the processes that occur in the region, serving as the basis for future work and monitoring of Mangueira Lagoon.

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