International Journal of Research (IJR)

e-ISSN: 2348-6848
p-ISSN: 2348-795X
Vol. 9 Issue 11
November 2022

Determination of lead in water from reservoirs for human supply using atomic absorption spectrometry

Douglas G. da Silva[1*], Darlene P. dos Santos[1], Evellyn C. G. Souza[1], Sulene A. Araújo[1], Augusto C. M. Aleluia[1]

*[1]Department of Natural Sciences, Laboratory of Analytical Chemistry, State University of Southwest Bahia – UESB; *douglas.goncalves@uesb.edu.br*

Abstract

This paper describes a procedure for the determination of Pb in water by employing a solid-phase preconcentration system using flame atomic absorption spectrometry (F AAS). The procedure was based on percolation of the sample onto a polyurethane foam support loaded with the complexing agent 2-(6'-methyl-2'-benzothiazolylazo)chromotropic (Me-BTANC). The applied method showed a linearity range from 5.0 to 30 µg L^{-1} and detection and quantification limits of 1.32 and 4.23 µg L^{-1}, respectively, with excellent precision and accuracy. The mean concentration of Pb quantified in waters of the Anajé-BA reservoir was 12.00 µg L^{-1} for the rainy period, while for the dry period the mean concentration found was 9.10 µg L^{-1}.

Keywords: *contamination, toxic metal, water, preconcentration.*

1. Introduction

Environmental contamination by toxic metals has generated concern throughout the scientific community and society in general. This concern is justified by the fact that these contaminants are non-biodegradable, bioaccumulative and highly toxic [1].

Received: *29 Oct. 2022*
Revised: *13 Nov. 2022*
Final Accepted for publication: *29 Nov 2022*

e-ISSN: 2348-6848
p-ISSN: 2348-795X
Vol. 9 Issue 11
November 2022

Among these species, lead (Pb) stands out for being abundant and dangerous in the form of cations or when bound to short carbonic chains [2].

The main way that lead is introduced into the environment is through the contamination of water bodies by anthropogenic activities such as: discharge of municipal effluents, metallurgical activities, mining and use of phosphate fertilizers in agriculture [3].

Phosphate fertilizers have been widely used in agriculture, since the low availability of phosphorus often limits the yield of crops under Brazilian conditions. These fertilizers, however, constitute a way for toxic metals to enter the soil [4].

In this context, there is a need for highly sensitive analytical methods capable of quantifying lead at low concentrations with high reliability. These methods, besides contributing to the quality control and preservation of water resources, can also help in the understanding of the propagation of lead in the environment and, additionally, be useful for the identification of pollutant sources. Pre-concentration, as an initial step in the analytical process, is necessary when the concentration of the analyte in the sample is below or close to the detection limit of the determination method. Among the various pre-concentration techniques used in the quantification of metals at trace levels, chemical sorption on modified solid supports has been widely used in recent decades [5].

Polyurethane foam presents excellent characteristics as a sorbent in solid phase extraction [6]. In recent years, this sorbent has been widely used in preconcentration systems for the determination of metals by flame atomic absorption spectrometry [7], [8].

Atomic absorption spectroscopy (AAS) has been a widely employed analytical technique to quantify toxic metals with high precision and sensitivity.

Received: *29 Oct. 2022*
Revised: *13 Nov. 2022*
Final Accepted for publication: *29 Nov 2022*

e-ISSN: 2348-6848
p-ISSN: 2348-795X
Vol. 9 Issue 11
November 2022

Given the above, this work aimed to determine the concentration of lead in the Anagé-BA water reservoir, through seasonal evaluations, employing preconcentration in solid phase for sample preparation and subsequent determination by flame atomic absorption spectrometry.

2. Material and methods

2.1 Reagents and solutions

All glassware, bottles, and materials used in the analytical procedures were washed with neutral detergent and immersed in 10% (v/v) HNO_3 solution for at least 24 h for decontamination. After this period they were washed with ultrapure water three times and air-dried in a dust-free environment.

The reagents used were of high purity and the solutions were prepared with ultrapure water obtained from a system (ELGA, Purelab classic) with a specific resistivity of 18 MΩ cm^{-1}.

The lead reference solutions were prepared in 5% (v/v) HNO_3 by dilutions of the 1000 mg L^{-1} stock solution (Fluka, Analytical).

2.2 Sampling and Sample Preparation

The sampling occurred seasonally in order to relate the behavior of water bodies and the concentration of lead throughout the dry and rainy periods. According to the data from the historical series of rainfall stations from the National Institute of Meteorology - INMET, the rainy period in the municipality of Anajé-BA corresponds to September 20 to April 30. The dry season corresponds from April 30 th to eptember 20th.

The samples were collected in triplicates, in the morning shift, and stored in Styrofoam boxes at an average temperature of 4 °C for further analysis.

Received: *29 Oct. 2022*
Revised: *13 Nov. 2022*
Final Accepted for publication: *29 Nov 2022*

e-ISSN: 2348-6848
p-ISSN: 2348-795X
Vol. 9 Issue 11
November 2022

Samples were collected and analyzed in the first and second semester of the year 2021 (rainy/dry period), in 4 points distributed along the Anajé-BA water reservoir. The samples were filtered through 0.45 µm cellulose membranes, acidified with HNO_3 1% (v/v) and stored at low temperatures.

2.3 Instrumentation

Lead determination was conducted in a flame atomic absorption spectrometer (FAAS), model AAnalyst 200 (USA) with an air/acetylene flame at flow rates of 3.5 L min^{-1} and 1.5 L min^{-1}.

The instrument response was checked periodically with standard solutions of known concentration. The data acquisition system was managed by a microcomputer coupled to the atomic absorption and, as a radiation source, a hollow cathode lamp of the element to be determined operating at 283.3 nm was used.

2.4 Preparation of the minicolumn

The minicolumn for lead preconcentration was prepared according to the method described by Gama, Da Silva and Lemos, with modifications [5].

A 4.5 cm long, 4.0 mm internal diameter serum tube was filled with approximately 100 mg of ground polyurethane foam. Then an alcoholic solution of 2-(6'-methyl-2'-benzothiazolylazo)chromotropic (Me-BTANC) 8.0 $x10^{-4}$ mol L^{-1} was percolated at a flow rate of 2.0 mL min^{-1} for 5 minutes using a peristaltic pump (Milan - Model P200). Then, the excess of Me-BTANC was eliminated using a 1mol L^{-1} NaOH solution. Subsequently, a solution of HNO_3 1 mol L^{-1} and water at the same flow rate was passed through the minicolumn to avoid contamination of other metals [9]. The minicolumn was constructed according to Figure 1.

Received: *29 Oct. 2022*
Revised: *13 Nov. 2022*
Final Accepted for publication: *29 Nov 2022*

e-ISSN: 2348-6848
p-ISSN: 2348-795X
Vol. 9 Issue 11
November 2022

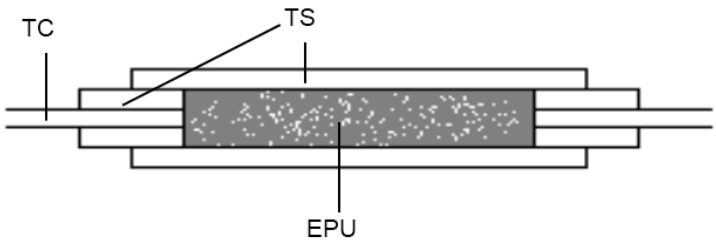

Figure 1: Type of minicolumn used in lead preconcentration. TG: silicone tube; TC: polytetrafluoroethylene capillary tube; EPU: polyurethane foam.

To construct the calibration curve, lead solutions with known concentrations between 0 and 30 µg L^{-1}, in borate buffer pH 7.5, passed through the minicolumn at a flow rate of 2 mL min^{-1}. Then, the lead retained in the column was eluted using HCl 1.0 mol L^{-1} at a flow rate of 2 mL min^{-1}, collecting 1.0 mL of the eluate for determination by atomic absorption spectrometry (F AAS). The preconcentration system used for the samples was operated by passing 25 mL of the sample using the same parameters as for the lead standard solutions.

3. Results and discussion

In the developed preconcentration system, lead showed a linearity range from 5.0 to 30 µg L^{-1} and limit of detection and quantification of 1.32 µg L^{-1} and 4.23 µg L^{-1}, respectively, calculated as recommended by IUPAC [10]. The method precision determined for lead concentrations of 5.0 and 20 µg L^{-1} for (n=7), was shown to be 1.2 and 0.81%, respectively. The accuracy was evaluated by determining lead in certified reference material, NIST SRM 1643d trace elements in natural water, which has a certified value for lead concentration of 18.15 ± 0.64 µg L^{-1}. From the applied method, the determined lead concentration was 17.92 ± 0.53 µg L^{-1}. Statistical comparison by

Received: *29 Oct. 2022*
Revised: *13 Nov. 2022*
Final Accepted for publication: *29 Nov 2022*

e-ISSN: 2348-6848
p-ISSN: 2348-795X
Vol. 9 Issue 11
November 2022

t-test showed no significant difference between the lead values obtained by the applied method and the certified value.

Thus, the method was used for lead determination in water samples from Anajé-BA reservoir. The mean concentration of lead quantified was 12.00 µg L^{-1} for the rainy season, while for the dry season the mean concentration was 9.10 µg L^{-1}. In Table 1 are presented, the concentrations of lead in the samples from each point, along with the respective standard deviation values.

The results showed that samples P1 and P2 have lead concentrations below the limit established by Health Organization Brazilian Govern equivalent to 10 µg L^{-1} [11]. However, samples P3 and P4 presented concentration values higher than the allowed limit. It was observed that the sample with the lowest concentration of lead in the dry period, also presented the lowest concentration of lead in the rainy period. The same pattern was observed for higher concentrations, which shows the persistence of lead in the environment.

Table 1: Lead concentration in water samples analyzed in the dry and rainy periods.

Sample	Pb concentration (µg L^{-1})	
	Dry period (June 2021)	**Rainy period (October 2021)**
P1	5.45 ± 0.71	8.65 ± 0.46
P2	7.72 ± 0.68	9.12 ± 0.51
P3	10.89 ± 0.52	14.69 ± 0.63
P4	12.36 ± 0.56	15.56 ± 0.48

Received: *29 Oct. 2022*
Revised: *13 Nov. 2022*
Final Accepted for publication: *29 Nov 2022*

e-ISSN: 2348-6848
p-ISSN: 2348-795X
Vol. 9 Issue 11
November 2022

The presence of lead found in the samples can be associated with natural processes and also be attributed to anthropogenic actions. Due to its capacity of bioaccumulation, this metal can enter the food chain and reach humans, causing damage to several organs, such as kidneys and nervous system [12]. Thus, its levels should be under constant monitoring.

In the field analysis, specifically in points P3 and P4, it is noted the presence of elements that have potential for lead release, such as chemicals and domestic effluent emissions. Besides these elements, along the collection points P3 and P4, there is a road where there is vehicle circulation, which may contribute to the increase in lead concentration.

4. Conclusions

With the pre-concentration procedure used, it was possible to determine lead in water samples. It can be concluded that the metal concentration found in the analyzed samples has a relevant influence of the seasonality. The lead concentration was high and above the values defined by the legislation in specific points of the Anajé-BA reservoir, indicating the need for constant monitoring.

Reference

[1] Ahmad, K., Wajid, K., Khan, Z. I., Ugulu, I., Memoona, H., Sana, M., Nawaz, K., Malik, I. S., Bashir, H., & Sher, M. (2019). Evaluation of Potential Toxic Metals Accumulation in Wheat Irrigated with Wastewater. Bulletin of Environmental Contamination and Toxicology, 102(6), 822–828. https://doi.org/10.1007/s00128-019-02605-1

Received: *29 Oct. 2022*
Revised: *13 Nov. 2022*
Final Accepted for publication: *29 Nov 2022*

e-ISSN: 2348-6848
p-ISSN: 2348-795X
Vol. 9 Issue 11
November 2022

[2] Yazdanfar, N., Vakili Saatloo, N., & Sadighara, P. (2022). Contamination of potentially toxic metals in children's toys marketed in Iran. Environmental Science and Pollution Research, 29(45), 68441–68446. https://doi.org/10.1007/s11356-022-20720-1

[3] Davenport, J. R., & Peryea, F. J. (1991). Phosphate fertilizers influence leaching of lead and arsenic in a soil contaminated with lead arsenate. Water, Air, and Soil Pollution, 57–58(1), 101–110. https://doi.org/10.1007/bf00282873

[4] Zhou, S., Liu, J., Xu, M., Lv, J., & Sun, N. (2015). Accumulation, availability, and uptake of heavy metals in a red soil after 22-year fertilization and cropping. Environmental Science and Pollution Research, 22(19), 15154–15163. https://doi.org/10.1007/s11356-015-4745-7

[5] Gama, E., Da Silva L. A., Lemos, V. A. (2006). Preconcentration system for cadmium and lead determination in environmental samples using polyurethane foam/Me-BTANC. Journal of Hazardous Materials, 136(3), 757–762. https://doi.org/10.1016/j.jhazmat.2006.01.015

[6] Braun, T., Navratil, J. D., & Farag, A. B. (2018). Polyurethane Foam Sorbents. Polyurethane Foam Sorbents in Separation Science, 1–157. https://doi.org/10.1201/9781351075916-1

[7] dos Santos, W. L., dos Santos, C. M. M., Costa, J. L. O., Andrade, H. M. C., & Ferreira, S. L. C. (2004). Multivariate optimization and validation studies in on-line

Received: *29 Oct. 2022*
Revised: *13 Nov. 2022*
Final Accepted for publication: *29 Nov 2022*

e-ISSN: 2348-6848
p-ISSN: 2348-795X
Vol. 9 Issue 11
November 2022

pre-concentration system for lead determination in drinking water and saline waste from oil refinery. Microchemical Journal, 77(2), 123–129. https://doi.org/10.1016/j.microc.2004.02.006

[8] dos Santos, W. N. L., Santos, C. M. C., & Ferreira, S. L. C. (2003). Application of three-variables Doehlert matrix for optimisation of an on-line pre-concentration system for zinc determination in natural water samples by flame atomic absorption spectrometry. Microchemical Journal, 75(3), 211–221. https://doi.org/10.1016/s0026-265x(03)00099-7

[9] Ferreira, S. L. C., Lemos, V. A., Moreira, B. C., Costa, A. C. S., & Santelli, R. E. (2000). An on-line continuous flow system for copper enrichment and determination by flame atomic absorption spectroscopy. Analytica Chimica Acta, 403(1–2), 259–264. https://doi.org/10.1016/s0003-2670(99)00632-7

[10] IUPAC, Analytical Chemistry Division, Spectrochim. Acta: Part B, 33 (1978) 242.

[11] Health ministry from Brazil, Ministério da Saúde, Portaria 518, 2004.

[12] Wang, J., Ren, T., Han, Y., Zhao, Y., Liao, M., Wang, F., & Jiang, Z. (2015). The effects of dietary lead on growth, bioaccumulation and antioxidant capacity in sea cucumber, Apostichopus japonicus. Environmental Toxicology and Pharmacology, 40(2), 535–540. https://doi.org/10.1016/j.etap.2015.08.012

Received: *29 Oct. 2022*
Revised: *13 Nov. 2022*
Final Accepted for publication: *29 Nov 2022*