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Tramonte *et al.* (2016) - Evaluation of metals availability in sediments of the Bertioga Channel (Santos Estuarine Complex -SP - Brazil): A tool for chemical pollution monitoring. *Journal of Integrated Coastal Zone Management / Revista de Gestão Costeira Integrada*, 16(X):xxx-xxx. DOI: 10.5894/rgci670 Supporting Information

Supporting Information I

Sequential extraction methodology for metals

Synthesis of sequential extraction procedure based on the protocol developed and optimized by the European Community Bureau of Reference (BCR) (currently named Standards, Measurements and Testing (SM&T) Programme) described in Pueyo *et al.* (2001):

- Step 1: Acid-soluble fraction (F1) 20 mL acetic acid 0.11 M was added for each 0.5 g of sediment sample. This suspension was agitated for approximately 16 h at room temperature and then centrifuged in order to separate the liquid extract and the solid phase.
- Step 2: Reducible fraction (F2) 20 mL hydroxylamine hydrochloride 0.5 M was added to the solid residue from step 1. Agitation, centrifugation and separation of the solid and liquid phases were performed according to the described on step 1.
- Step 3: Oxidizable fraction (F3) 5 mL hydrogen peroxide 30% (v/v) was added to the solid residue from step 2. Initially, the extraction was performed at room temperature. Afterwards, another aliquot of 5 mL hydrogen peroxide was added under heating. After reducing the volume and cooling of the suspension, it was added 25 mL ammonium acetate 1 M (pH = 2) to it. Agitation, centrifugation and separation of the solid and liquid phases were performed according to the described on step 1.

Those above-described extractions were performed with dry sediment and the residues were washed with Milli-Q water, under agitation, before the use of the aliquots on the subsequent steps. The liquid extracts obtained on each step were kept under refrigeration until the moment of analysis. Figure SI.1 shows the flowchart of this methodology steps.











Figure SI.1 - Scheme of sequential extraction for metals based on BCR method

Supporting Information II

Metal content in acid-soluble fraction (F1), reducible fraction (F2) and oxidizable fraction (F3)

Figures SII.1 to SII.25 show graphically the metal content in each mobile fraction, as well as the available content (F1 + F2 + F3) in the sample. Absence of the content of one or more fractions means that the value was below the quantification limit (QL) of the element.

a) Cr



Figure SII-1 - Content of Cr (in mg kg⁻¹) associated with the mobile fractions in B1 sediment core of the Bertioga Channel.



Figure SII-2 - Content of Cr (in mg kg⁻¹) associated with the mobile fractions in B2 sediment core of the Bertioga Channel.



Figure SII-3 - Content of Cr (in mg kg⁻¹) associated with the mobile fractions in B3 sediment core of the Bertioga Channel.



Figure SII-4 - Content of Cr (in mg kg⁻¹) associated with the mobile fractions in B4 sediment core of the Bertioga Channel.



Figure SII-5 - Content of Cr (in mg kg⁻¹) associated with the mobile fractions in B5 sediment core of the Bertioga Channel.





Figure SII-6 - Content of Cu (in mg kg⁻¹) associated with the mobile fractions in B1 sediment core of the Bertioga Channel.



Figure SII-7 - Content of Cu (in mg kg⁻¹) associated with the mobile fractions in B2 sediment core of the Bertioga Channel.



Figure SII-8 - Content of Cu (in mg kg⁻¹) associated with the mobile fractions in B3 sediment core of the Bertioga Channel.



Figure SII-9 - Content of Cu (in mg kg⁻¹) associated with the mobile fractions in B4 sediment core of the Bertioga Channel.



Figure SII-10 - Content of Cu (in mg kg⁻¹) associated with the mobile fractions in B5 sediment core of the Bertioga Channel.



Figure SII-11 - Content of Ni (in mg kg⁻¹) associated with the mobile fractions in B1 sediment core of the Bertioga Channel.



Figure SII-12 - Content of Ni (in mg kg⁻¹) associated with the mobile fractions in B2 sediment core of the Bertioga Channel.



Figure SII-13 - Content of Ni (in mg kg⁻¹) associated with the mobile fractions in B3 sediment core of the Bertioga Channel.



Figure SII-14 - Content of Ni (in mg kg⁻¹) associated with the mobile fractions in B4 sediment core of the Bertioga Channel.



Figure SII-15 - Content of Ni (in mg kg⁻¹) associated with the mobile fractions in B5 sediment core of the Bertioga Channel.



d) Pb

Figure SII-16 - Content of Pb (in mg kg⁻¹) associated with the mobile fractions in B1 sediment core of the Bertioga Channel.



Figure SII-17 - Content of Pb (in mg kg⁻¹) associated with the mobile fractions in B2 sediment core of the Bertioga Channel.



Figure SII-18 - Content of Pb (in mg kg⁻¹) associated with the mobile fractions in B3 sediment core of the Bertioga Channel.



Figure SII-19 - Content of Pb (in mg kg⁻¹) associated with the mobile fractions in B4 sediment core of the Bertioga Channel.



Figure SII-20 - Content of Pb (in mg kg⁻¹) associated with the mobile fractions in B5 sediment core of the Bertioga Channel.

e) Zn



Figure SII-21 - Content of Zn (in mg kg⁻¹) associated with the mobile fractions in B1 sediment core of the Bertioga Channel.



Figure SII-22 - Content of Zn (in mg kg⁻¹) associated with the mobile fractions in B2 sediment core of the Bertioga Channel.



Figure SII-23 - Content of Zn (in mg kg⁻¹) associated with the mobile fractions in B3 sediment core of the Bertioga Channel.



Figure SII-24 - Content of Zn (in mg kg⁻¹) associated with the mobile fractions in B4 sediment core of the Bertioga Channel.



Figure SII-25 - Content of Zn (in mg kg⁻¹) associated with the mobile fractions in B5 sediment core of the Bertioga Channel.

Reference

Pueyo, M., Rauret, G., Lück, D., Yli-Halla, M., Muntau, H., Quevauviller, Ph., López-Sánchez, J.F. (2001) - Certification of the extractable contents of Cd, Cr, Cu, Ni, Pb and Zn in a freshwater sediment following a collaboratively tested and optimised three-step sequential extraction procedure. Journal of Environmental Monitoring, 3(2):243-250. DOI: <u>10.1039/b010235k</u>.