A PRELIMINARY STUDY OF LIPOPHILIC TOXINS IN GALICIAN SEDIMENTS

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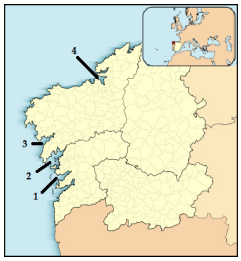
ABSTRACT

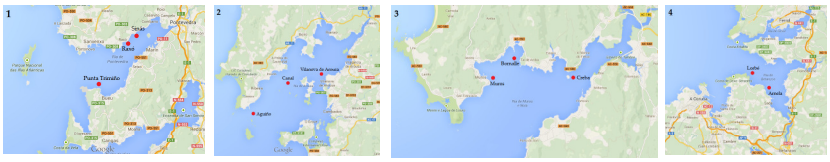
For decades, okadaic acid has been broadly detected in Galician Rías causing frequent bans of shellfish harvesting. In addition, the presence of 13-desmethyl SPX C has been commonly recorded in seawater and shellfish from the same area since LC-MS/MS started to used routinely to check the presence of lipophilic toxins. This work tryes to elucidate if sediment profiles could be an useful tool to study the incidence of harmful events in the past. For that reason, sediment samples from different locations of the Galician coast (rías of Ares-Betanzos, Arosa, Muros and Pontevedra) were analyzed by LC-MS/MS in order to evaluate the presence of lipophilic toxins in sediments. The method includes the following toxins: yessotoxin; okadaic acid; DTX-1; PTX-2; 13 desmethyl SPX C; 13, 19 didesmethyl SPX C; Pinnatoxin F and Pinnatoxin G, Gymnodimine, azaspiracids 1-5, and the tentative identification of other toxins. 13 desmethyl SPX C, okadaic acid and PTX-2 were often detected above the LOQ of the LC-MS/MS method in all the studied Rías. Levels of 13 desmethyl SPX C are slightly higher than those of PTX-2. Okadaic acid is the toxin that reaches the highest concentrationin some samples maximum concentration was found in subsurficial sediments (Arosa, Muros and Pontevedra Rías).

OBJECTIVE

Evaluate the concentration and persistence of marine biotoxins in Galician sediments to assess the influence of toxic events in the past.

METHODS





Cores in mussel culture areas (rafts) → around 40-120 years

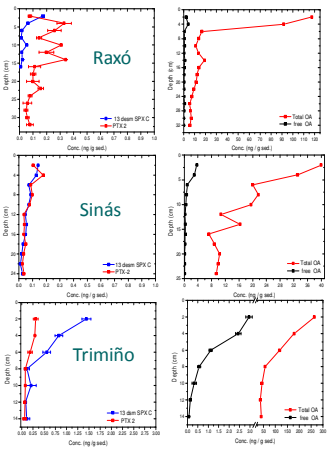
Row Extracts:

1g sed. → MeOH 100% (1/4 p:v) → 11000rpm 3' → Centrifuge (48000g 15') → 0.2 µm filter → LC-ESI-MS/MS (MRM method).

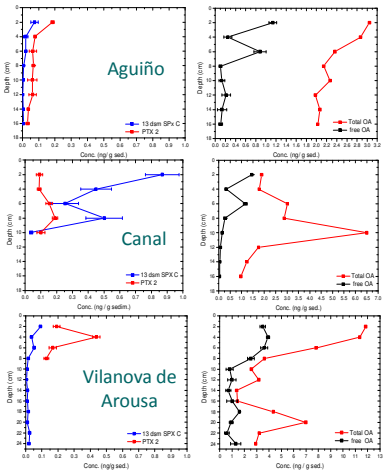
Hidrolysis: 1mL of row extract + 125 µL NaOH 2.5M → 76ºC 40' → +125µL HCl 2.5M.

RESULTS

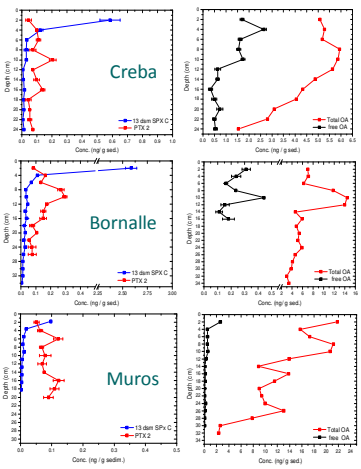
Ría de Pontevedra



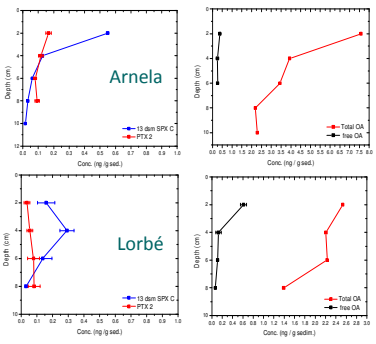
Ría de Arousa



Ría de Muros



Ría de Ares



CONCLUSIONS

- The most common toxins in Galician sediments are 13 dsm SPX C, OA and PTX-2.
- Okadaic acid in sediments is mainly found in conjugated form.
- The highest OA concentrations were found at superficial sediments → probably increasing of harmful event during last decades.
- Some subsuperficial maxima of 13 dsm SPX C were found in some areas suggesting that some in some places it could had have more importance that at present.
- PTX-2 and 13 dsm SPX C are present in low concentrations at sediments probably because degradation.
- PTX-2 and OA total concentration are related as it could be expected from the development of Dinophysis populations

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