Toxicity of coastal waters: use of a quick algal bioassay

Citation for published version (APA):

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SETAC Europe 21st Annual Meeting

Ecosystem Protection in a Sustainable World:
A Challenge for Science and Regulation

15–19 May 2011

abstract book
choice of sorbent is crucial in SPE because it can control such parameters as selectivity, affinity and capacity. This choice depends strongly not only on the target analyte and the interactions of the chosen sorbent through the functional groups of the analytes, but also on the kind of sample matrix and its interactions with both the sorbent and the analytes. This work describes the application of the different kinds of SPE sorbents: C18 bonded silica gel (Strat C18, C18), polymers (Osiris HLB, Strata X, and LiChrosor FN), functionalized polymers (Isolute ENV+), mixed-mode sorbents (phenyl/polymer MCX) and a three-function sorbent (Strata Screen C) for extraction of six β-blockers (acetylbutol, atenolol, metoprolol, nadolol, propranolol, pindolol, and two β-adrenoeceptors (terbutaline, salbutamol) from natural water samples. Parameters such as pH of the loading samples, the amount and the kind of solvents used in conditioning, washing and eluting steps, were selected and optimized. The obtained extracts were evaporated to dryness, subjected to silylation using BSTFA, and finally analysed by GC-FID technique. The recovery of the analytes form natural water samples in the mentioned above SPE conditions will be discussed.

Acknowledgement: Financial support was provided by the Polish Ministry of Research and Higher Education under grant N N204 260237 (2009-2012).

TU 082

Musteep fractionation based on normal phase SPE and reverse phase HPLC (RP-HPLC) for isolation of endocrine disrupting chemicals in environmental extracts

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An Improved Directed Edaphic Analysis (EDA) approach aims to identify adverse pollutants by reducing the complexity of environmental matrices. Single hyperfractionation combined to bioassays is capable to link specific active chemicals and to direct unknown hyperfractionation steps to these “classical” pollutants. However, although the promise of emerging promising chemical tools (e.g. Orbitrap1), identification of unknown active chemicals is still time and cost consuming due to the complexity of each active fraction (e.g. mixture effect). Hence, further fractionation steps are often needed. The aim of this study was to develop and to test the use of a first pre-fractionation step on SPE that will be followed by a RP-HPLC fractionation. First the separation of 12 EDG’s have been evaluated with several elution conditions. Silica cartridges with 4 step elution - heptane, heptane/dichloromethane (50/50, v/v), ethyl-acetate and methanol/water (50/50, v/v) - were used. The effect of the elution order, the number of blocks, have been chosen for further investigations. For these conditions, recoveries were assessed for the mixture alone and for a blank sediment extract spiked with this mixture. Finally, a natural sediment known to exert estrogic, PXR- and CAR-like activity in vitro and in vivo. Good mixture recoveries (74-110 %), were obtained. The fractionation F1 contained only the PCBs and the PAHs, while 4-tert-octylphenol, triphenyl phosphate and fenofibrate were detected only in F2. Finally, steroids, bisphenol A and clomifene were found in F3 while F4 contained more polychlorinated chemicals. Fractionation on natural sediment allows isolation of TCDD-like activity in F1 and F2 while PAH like activity was detected in F1, F2 and in F3. Then estrogenic compounds were only detected in F2 and F3. Interestingly, the sum of the estrogenic activity found in these 2 fractions is higher than the activity of the crude extract, whereas the occurrence of anti-estrogenic chemicals. Finally, PXR-like activity was mainly detected in F3. This pre-fractionation protocol allows, in the present case study, the isolation of several biological activities. Based on this first isolation directed hyperfractionation has then been undergone, RP-HPLC (hyperfractionation on basis MCX) and a three-function sorbent (Strata Screen C) allows the storage of a wide variety of spectra including EI-MS, ESI-QTOF-MSMS and ESI-FTI-MS. Different tools are available to process the raw data and upload the data to Massbank including a spreadsheet based record editor for the addition of metadata.

References:

TU 084

Construction of a water toxicity sensor based on luminescent bacteria

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Microcystis, highly toxic cyclic peptides, are a group of hepatotoxins produced by a number of aquatic species of cyanobacteria, such as Microcystis, Anabaena and Planktothrix. Worldwide, concentrations in water have increased over the past decades, in response to eutrophication and increased levels of nutrient availability. World wide, cyanobacteria blooms are increasing in numbers and in complexity of each active fraction (e.g. mixture effect). Hence, further fractionation steps are often needed. The aim of this study was to develop and to test the use of a first pre-fractionation step on SPE that will be followed by a RP-HPLC fractionation. First the separation of 12 EDG’s have been evaluated with several elution conditions. Silica cartridges with 4 step elution - heptane, heptane/dichloromethane (50/50, v/v), ethyl-acetate and methanol/water (50/50, v/v) - were used. The effect of the elution order, the number of blocks, have been chosen for further investigations. For these conditions, recoveries were assessed for the mixture alone and for a blank sediment extract spiked with this mixture. Finally, a natural sediment known to exert estrogic, PXR- and CAR-like activity in vitro and in vivo. Good mixture recoveries (74-110 %), were obtained. The fractionation F1 contained only the PCBs and the PAHs, while 4-tert-octylphenol, triphenyl phosphate and fenofibrate were detected only in F2. Finally, steroids, bisphenol A and clomifene were found in F3 while F4 contained more polychlorinated chemicals. Fractionation on natural sediment allows isolation of TCDD-like activity in F1 and F2 while PAH like activity was detected in F1, F2 and in F3. Then estrogenic compounds were only detected in F2 and F3. Interestingly, the sum of the estrogenic activity found in these 2 fractions is higher than the activity of the crude extract, whereas the occurrence of anti-estrogenic chemicals. Finally, PXR-like activity was mainly detected in F3. This pre-fractionation protocol allows, in the present case study, the isolation of several biological activities. Based on this first isolation directed hyperfractionation has then been undergone, RP-HPLC (hyperfractionation on basis MCX) and a three-function sorbent (Strata Screen C) allows the storage of a wide variety of spectra including EI-MS, ESI-QTOF-MSMS and ESI-FTI-MS. Different tools are available to process the raw data and upload the data to Massbank including a spreadsheet based record editor for the addition of metadata.

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