Toxicity of coastal waters: use of a quick algal bioassay


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Environmental water sample matrices, especially sewage and marine-water samples are complex and often contain interfering elements that can mask or interfere with the analysed pharmaceutics. This work describes the application of different kinds of SPE sorbents: C18 bonded silica gel (Strata C18), copolymers (OS40 HLB, Strata X, and Lichrolut EN), functionalised copolymers (Isolute ENV+), mixed-mode (strong anion exchange and CMC) and Alberta bitumen (Strata Sorbent C60) for extraction of six β-blockers (acebutolol, atenolol, metoprolol, nadolol, propranolol, pindolol,) and two β-,-,- and 6-aminodioxin (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 by BSTFA, and finally analysed by GC-FID technique. The recovery of the analyses form natural water samples in the mentioned above conditions will be discussed.

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TU 082

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

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Endocrine Disrupted (EDA) approach aims to identify adverse pollutants by reducing the complexity of environmental matrices. Single hyperfractionation combined to biosays is useful to find active chemicals and to direct chemical analyses to these "classical" pollutants. However, although the emergence of promising chemical tools (e.g. Orgasorb), 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 EDCs 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 best results for the isolation of PAHs, which 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 estrogenic, PXR-like and anti-androgenic activity in radial in vitro tests conditions. 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 clortimazole were found in F3 while F4 contained more polar 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 measured in the crude extract, which indicates 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 (RP-HPLC) and a three-function assay (CMC) and a function analysis is on progress for further investigations. This work is a proof of principle for a first step of a pre-fractionation process which allows, in the case of endocrine disruptors, the isolation of each fraction on chemical properties and will be readily used for the isolation of active chemicals in the polar and semi-polar pre-fractions.

TU 083

Towards a common mass spectral database for the identification of unknowns in environmental samples

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Environmental matrices, especially sewage and marine-water samples are complex and open access mass spectra database including MS data from all instrument types and with soph-...