Toxicity of coastal waters: use of a quick algal bioassay


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TU 081
Optimization of the SPE step in the analysis of β-blockers and β-adrenoeceptors in natural water samples by SPE–GC technique
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Environmental sampling techniques, especially sewage and marine water samples are complex and often contain interfering elements that can mask or interfere with the analysed pharmaceu-
ticals. Therefore, it is important to optimize the SPE step for the analysis of these compounds. The solid phase extraction (SPE) is the most common sample preparation technique used in environmental areas. Choice of sorbent is a crucial in SPE because it can control such parameters as selectivity, affinity and capacity. This choice depends strongly not only on the target analytes 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 ap-
plication of the different kinds of SPE sorbents: C8 bonded silica gel (Strata C18), copolymers (Octisil HLB, Strata X, and Lichrolut EN), functionalised copolymers (Isolute ENV+), mixed-
mode sorbents (Strata Screen C), and a three-function sorbent (Strata Screen C). For extraction of six β-blockers (acebutolol, 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 silution by 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.

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Butterstep 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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Directed Extracted Analysis (DEA) approach aims to identify adverse pollutants by reducing the complexity of environmental matrices. Single hyperfractionation combined to bioas-
say is helpful to isolate active chemicals and to direct chemical analyses towards these "classical" pollutants. However, although the emergence of promising chemical tools (e.g. Or-
bitrap), 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 aims 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)) have been used for the isolation of these compounds, 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 activities was fractionated following these 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, benzo(a)anthracene and chlorzamol 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 PAF like activity was detected in F1, F2 and in F3. Then estrogenic compounds were only de-
tected in F2 and F3. Interestingly, the sum of the estrogenic activity found in these 2 fractions is higher than the activity detected in the crude extract, which might indicate an occurrence of anti-estrogenic chemicals. Finally, PXR-like activity was mainly detected in F3.

Difficulties in characterizing endocrine active material are responsible for the toxic effect on the algae. In 2010-2011 passive samplers are exposed at Hansweert (WESTERSCHELD, The Netherlands) and collected every 6 weeks to include episodic events into account. The concentrated extracts are tested in an algal bioassay with Pulse Amplified Modulation (PAM) fluorometry provides a quick (4 h) method to determine toxicity of algae based on changes in photophysiological ef-
ficiencies. The Effect Directed Analysis strategy will be performed to unravel which chemical compounds are responsible for the toxic effect on the algae. In 2010-2011 passive samplers are exposed at Hansweert (WESTERSCHELD, The Netherlands) and collected every 6 weeks to include the seasonal dynamics of both algal blooms as well as natural compounds. Here, first results of this sampling strategy are presented and discussed. The results of the EDA analysis will be used in experiments where mixture toxicity, multi stress and community effects are taken into account to describe the overall toxic effect under relevant field conditions.

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Reference:

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Construction of a water toxicity sensor based on luminescent bacteria
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