Integrated and Automated Sample Preparation to Monitor Small Organic Pollutants

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A fully automated and portable platform for on-site sample preparation is being developed within the EU project RADAR to monitor endocrine disruptive compounds in water and food processes. Solid phase extraction is performed on a microfluidic device that enables the manipulation of small volumes of solution and fast sample preconcentration. The method is suited for sample preparation for analysis using biodetection methods, from standard well-plate format to label-free optical biosensors.

Endocrine disrupting compounds (EDCs) are a family of pollutants originating mostly from the degradation of plastics, smokes and drugs. Today, it is proven that they cause feminization of aquatic species in contaminated lakes and rivers, which increases human health concerns. Within the European RADAR [1] (Rationally Designed Aquatic Receptors) project, the development of an integrated and automated sample preparation platform is required prior to optical label-free detection for on-site monitoring of those compounds.

The portable sample preparation system that is being developed performs fully automated liquid sample collection, filtration of particles, removal of gas bubbles, enrichment of the target compounds and delivery of the different buffer solutions and reactants to the biosensor.

Since EDCs are found at very low levels in the environment (< 1 ppb = 1 ng/ml), i.e. below the limit of detection (LOD) of most biosensors, a preconcentration step is required. Methods based on solid phase extraction (SPE) are the most efficient in terms of separation and enrichment. However, SPE techniques are adapted to detection by chromatography and mass spectrometry and imply the use of organic solvents through many time-consuming steps. For biosensing monitoring purposes, the speed of the process, availability of the resources and final buffer conditions are critical aspects.

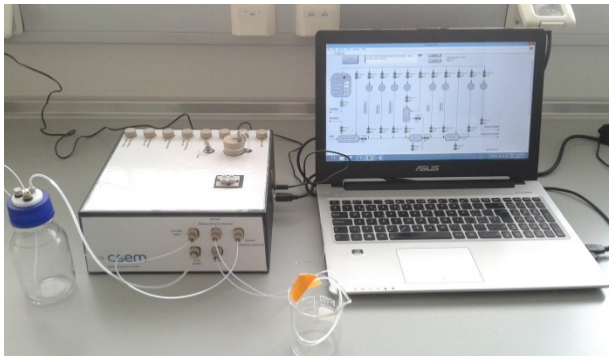

Figure 1: The sample preparation unit (SPU) as used in a laboratory.

A fully automated platform (Figure 1) was developed adapting and integrating a SPE method [2]. This system can be used independently or on-line with a biosensor. The main element of the instrument is the polyether ether ketone microfluidic device (Figure 2) that enables facile control of small fluid volumes.

Compared to traditional SPE procedures, our method requires smaller volumes of solution (3-30 ml sample, 20 µl eluent minimum), it is faster and user-friendly. Another key advantage is its full compatibility with immuno-detection methods by providing an enriched sample in an aqueous buffer with low solvent content. Finally, our instrument offers the possibility to adapt the parameters of the SPE procedure to provide the preconcentration factor required for any immunoassay.

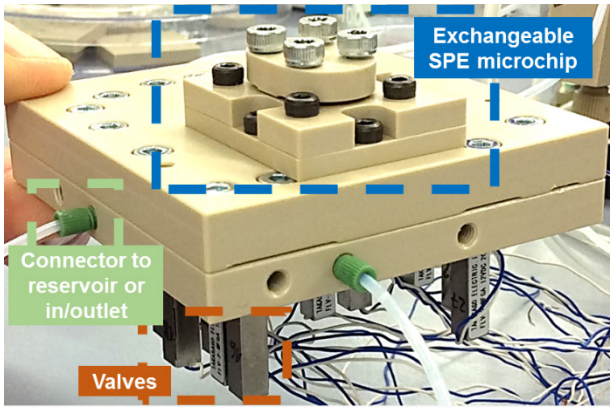

Figure 2: Picture of the PEEK microfluidic device (dimensions 7 x 6.5 cm long and 2.5 cm thick including the SPE microchip).

Our method was successfully applied to seawater samples spiked with 50 ng/l of 17β-estradiol. Our method was compared with a bench-top SPE procedure performed by the Rikilt Institute for Food Safety in the Netherlands. The molecule was detected using a label-free receptor assay on a surface plasmon resonance (SPR) biosensor. Satisfactory results were obtained with our instrument (Table 1), with a considerable reduction in the time needed for the sample preparation.

Table 1: Comparison of method and results obtained with the SPU and a bench-top procedure from RIKILT, with E2 in seawater samples, detected with a receptor assay on a SPR platform.

Method	Sample volume	SPE eluent	Time	Recovery
Bench-top	50 ml	1 ml solvent 100 vol%	≥ 2 h	68 %
SPU	30 ml	84 µl solvent 60 vol%	20 min	58 %

The next development steps will focus on the mechanical robustness of the instrument. The method will be applied to other EDCs and liquid sample matrices, such as drinking water, beer and milk.

[1] www.fp7-radar.eu

[2] S. Heub, L. Barbe, S. Follonier, P.S. Dittrich, "*Fully automated and portable platform for integrated extraction and pre-concentration of toxins and pollutants from liquid samples*", Proc. of the 18th micro-TAS conference, 2014.