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Time-integrated monitoring of dioxin-like polychlorinated biphenyls (dl-PCBs) in aquatic environments using the ceramic toximeter and the CALUX bioassay

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ABSTRACT

Ceramic passive samplers or toximeters (packed with active carbon 1%, w/w, on celite), in combination with the CALUX bioassay have been used as a time-integrated monitoring technique for dissolved dioxin-like PCBs in urban and industrial wastewaters. The technique showed to be reliable during laboratory experiments: (1) PCB-126 amounts extracted from the passive samplers increased linearly with the time of exposure and (2) PCB-126 concentrations calculated from the amounts accumulated by the passive samplers were in agreement with their concentrations in the testing solution. Afterwards the toximeters were applied in the field. Two sampling sites located in Egypt were chosen: the Belbeis drainage canal, and the EMAK paper mill. A total of 18 ceramic toximeters were exposed to the wastewater in both sampling sites for a maximum period of 4 weeks. Two samplers were collected weekly from each site to monitor the increase in target analytes over time. Extracts were analyzed using the CALUX bioassay and the total dioxin-like PCB toxicity was reported for the aqueous phase (water column), as well as the solid phase (sediment and sludge) in both sampling sites. The time-weighted average concentration (TWA) of dl-PCBs in wastewater of the paper mill during the sampling period ranged between 7.1 and 9.1 pg-BEQ L⁻¹, while that of the drainage canal ranged between 9.5 and 12.2 pg-BEQ L⁻¹. The dl-PCBs in the fibrous sludge (paper mill) and bottom sediment (drainage canal) were 0.5 and 0.4 pg-BEQ g⁻¹ dry-weight, respectively. The organic-carbon normalized partition coefficients between sediment and water (log K_{oc}) for the paper mill and the canal were 2.4 and 4.3, respectively.

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1. Introduction

The thermal and chemical stability of polychlorinated biphenyls (PCBs), among other properties, led to a widespread use in transformers, capacitors, hydraulic fluids, as well as plasticizers, and numerous other industrial applications [1]. Amongst the 209 possible PCB congeners, only 12 have dioxin-like toxicity, since they bind to the Ah-receptor [2,3]. Like most of the other PCB congeners, these dioxin-like PCBs have the potential to cause adverse effects on human and animal health [4]. In fact, the combined effect of their bioaccumulation in the trophic chain and the action of sediments as a reservoir of PCBs make the direct discharge of these compounds into the aquatic system problematic [5]. Their half-life time in these matrices can be as long as several

decades [6,7]. Since the major pathway of human exposure to dioxin-like compounds (over 90%) is through the diet [8], especially fish consumption [9,10], it is highly important to monitor dioxin-like PCBs in aquatic systems on a regular basis.

Ceramic-based passive sampling devices have been previously used for monitoring dissolved polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzofurans (PCDD/Fs) [11,12], polycyclic aromatic hydrocarbons (PAHs) and volatile organic contaminants (VOCs) in surface and ground water [13,14], and flame retardants in river water [15]. The ceramic toximeter is based on the free flow of analyte molecules from the sampled medium through a ceramic diffusion membrane towards a suitable receiving phase, under the effect of a chemical-potential difference [16]. This diffusion-controlled collection of analytes allows for calculating the time-weighted average (TWA) concentration of the target chemicals in the sampled medium during the sampling period depending on Fick's first law of diffusion. In this study a novel, time integrated monitoring method for dl-PCBs was validated. The analytical

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procedure, which is much faster and easier than earlier used methods, involves the in situ pre-concentration of dl-PCBs on activated carbon and their subsequent quantification by the CALUX bioassay. After validation, the toximeters were deployed in different wastewaters in Egypt.

2. Materials and methods

2.1. Reagents and materials

The ceramic cylinders were manufactured by ATECH Innovations (Germany) [12]. The XCARB (activated carbon 1%, w/w, on celite) was from Xenobiotic Detection Systems (USA). 3,3',4,4',5-Pentachlorobiphenyl (PCB-126, 99.4%) was from Chem Service (USA). Acetone (Pesti-S grade, minimum 99.9%), *n*-hexane (for dioxins and PCBs, minimum 96%) and toluene (for dioxins and PCBs, minimum 99.8%) were purchased from Biosolve (The Netherlands). Neutral alumina (activated, 150 mesh), silver nitrate (5 wt % on silica gel 60), ethyl acetate pestanal and silica gel 60 were purchased from Sigma–Aldrich (Germany). Anhydrous sodium sulfate was purchased from Boom (The Netherlands). Sulfuric acid (95–97%, ACS reagent), DMSO and glass wool were from Merck (Germany). Standard solution of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (50 µg/ml, in nonane) was from Campro Scientific (The Netherlands). The mouse hepatoma H1L7.5c1 cell line used in the CALUX bioassay was provided by Michael Denison (University of California, USA).

2.2. Conditioning of materials

The XCARB and glass wool for the toximeters were conditioned by fluxing in toluene as described previously [12]. The ceramic cylinders were sintered at 750 °C for 24 h to eliminate possible organic traces. PTFE caps and stainless steel cages were rinsed with acetone and Milli-Q deionized water. Silica gel was baked at 200 °C for 48 h prior to use. Glassware and sodium sulfate were baked for 4 h at 450 °C.

2.3. Laboratory testing of toximeters

A generated aqueous solution of PCB-126 was used as a stock solution in the laboratory testing experiments of the toximeters. Fifty micrograms of 3,3',4,4',5-pentachlorobiphenyl (PBC-126) were dissolved in 2 mL acetone, and then completed to 1000 mL with deionized water in a light-shielded glass bottle. The solution was shaken for 48 h at 40 rpm. The bottle cap was then removed under the hood for 72 h to allow a slow but full evaporation of acetone. A diluted PCB-126 testing solution was prepared for laboratory testing of the toximeters. Therefore, a 1 L glass bottle was filled with the testing solution for 48 h (prior to applying the toximeters) in order to equilibrate the PCB-126 between the solution and the glass wall. The drained solution is then replaced with a fresh amount of the same concentration. This, in turn, shall reduce the effect of PCB partitioning due to partial saturation of the bottle wall. At the start of the experiment ($t=0$ h), 8 toximeters were immersed in 1 L of the testing solution and after 12, 24, 48, and 96 h of exposure, 2 toximeters were removed from the solution. The bottle containing the toximeters was kept on a shaker (at 40 rpm) for the whole period of exposure.

To measure the concentration of this solution, 10 mL of the PCB solution was extracted 4 times with solvent (10, 10, 5, and 5 mL toluene, respectively). Toluene was evaporated and the extract was redissolved in 10 mL *n*-hexane and analyzed with the CALUX bioassay technique. Concentration of the testing solution at the start of the experiment ($t=0$ h) amounted to $0.78 (\pm 0.09) \mu\text{g L}^{-1}$

while at the end it decreased with about 50%. These concentrations were calculated from full dose–response curves (Fig. 1) of the PCB-126 testing solution and of 2,3,7,8-TCDD standard using a CALUX-REP value of 0.038 [17].

2.4. Field application of toximeters

Two wastewater sites located in Egypt were chosen as field sites: (1) the Belbeis drainage canal (N: 30°26'25.88", E: 31°34'23.16"), which is heavily polluted and frequently used to convey raw industrial and municipal wastewater and (2) the EMAK Paper Manufacturing Company (N: 29°41'50.12", E: 32°18'13.30"), Ain El-Soukhna, Suez. More details about the sampling locations can be found elsewhere [12]. Wastewater effluents of the paper mill were sampled by applying the toximeters before the primary wastewater treatment process inside the factory. Toximeters exposure continued for 4 weeks in October 2011. Every 7th day, two samplers were removed from each site. Bottom sediment from the drainage canal and fibrous sludge from the paper mill were analyzed to calculate the partitioning coefficient for dioxin-like PCBs in both sampling sites. The whole extraction, cleanup and analysis procedure is schematically presented in Fig. 2. More details are given below.

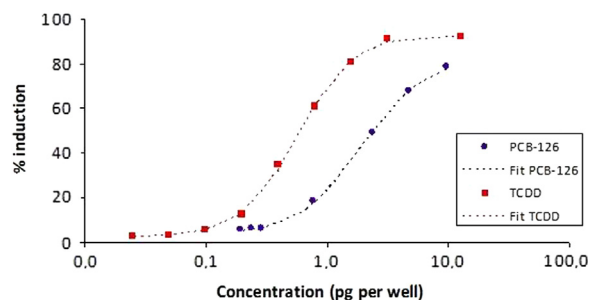


Fig. 1. Dose–response curves of a PCB-126 solution and a TCDD standard calibration curve.

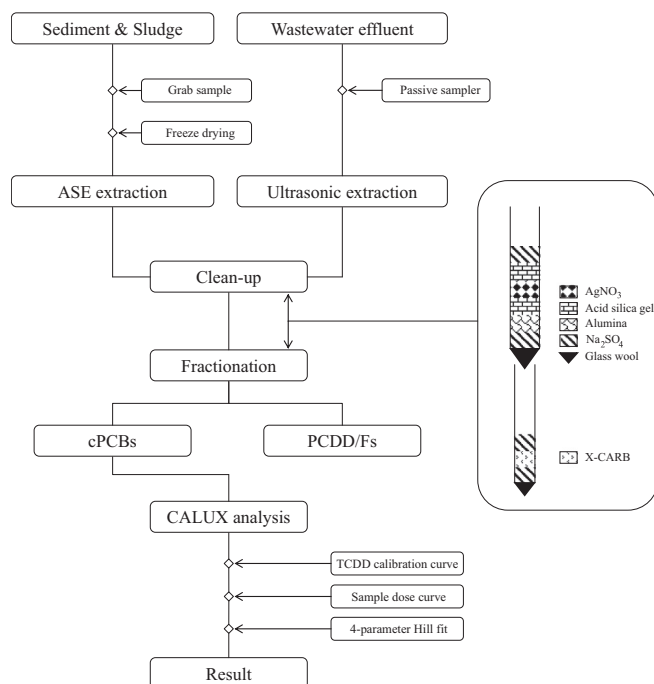


Fig. 2. Extraction, cleanup and analysis scheme of the CALUX bioassay procedure for dl-PCBs.

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