Detection of Perfluorocarboxylic Acids in Soil

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Naturally occurring fluorinated compounds tend to only contain one fluorine atom. This distinction makes it obvious that all other compounds containing two and up to a multitude of fluorine atoms are usually man-made[1]. Perfluoroalkyl substances (PFAS), as a group of compounds, amass over 4500 substances with varying structure numbers of fluorine atoms bound to a carbon backbone, of which approximately 25 % are registered with the US EPA Toxic Substances Control Act. Perfluoro carboxylic acids (PFCAs) in particular were produced by electrochemical fluorination from 1947 onwards[2]. The intrinsic properties of PFCAs, e.g., the hydrophobic and lipophobic behaviour, find application in a multitude of industries, posing an environmental risk globally.

However, detection of trace amounts of PFCAs in the environment still presents difficulties with environmentally friendly extraction practices and affordable and efficient detection in complex matrices. In this project we show the application of a sonication-assisted extraction procedure requiring minimal solvent usage for extracting PFCAs from biosolids and soil, which minimises the risk of cross-contamination. Biosolids and soil samples from agricultural land were collected over a period of two years, dried, processed, extracted and analysed before statistical evaluation.

The trace analysis of compounds was performed using LC-Orbitrap-MS. The nature of the setup allowed the analysis of PFBA (C4) up to PFDoA (C12) of perfluoro carboxylic acids. The analysis took place on a C18 Accucore (RP) column (100 mm x 2.1 mm x 2.6 µm) with a 22-minute gradient elution method. The MS detection in conjunction to LC was performed with an electrospay ionisation source (ESI) in line with other publications.

Preliminary results in soil showed the presence of PFHpA (C7, 1.1 – 22.2 ng/g), PFOA (C8, 2.7 ng/g), PFNA (C9, 0.1 – 0.7 ng/g), PFDA (C10, 0.1 – 1.8 ng/g) and PFDoA (C12, 0.7 ng/g). PFBA, PFPeA (C5), PFHxA (C6) and PFUnA (C11) were not detected in the analysis of the selected soil samples despite calibrations in the range of 0.1 to 100 ng/mL.

The comparison of biosolids and soil might allow for the tracing of PFAS from one complex matrix to the next. The presence of PFCAs in an agricultural frame, irrespective of the low-level concentrations, implicates the possibility of the introduction of contaminants into the food chain (e.g., the absorption into plant material (bioaccumulation), or the ingestion of soil and plants by livestock (biomagnification)) and should be investigated further.

1. Key BD, Howell RD, Criddle CS. Fluorinated Organics in the Biosphere. *Environ Sci Technol.* 1997;31:2445-2454.

2. Prevedouros K, Cousins IT, Buck RC, Korzeniowski SH. Sources, fate and transport of perfluorocarboxylates. *Environ Sci Technol.* 2006;40:32-44.