Detection of Perfluoro Carboxylic Acids in Complex Environmental Matrices

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The presence of Perfluoro carboxylic acids (PFCAs) in an agricultural frame, irrespective of the low-level concentrations, implicates the possible introduction of these contaminants into the food chain and needs to be investigated. PFCAs, produced by electrochemical fluorination from 1947 onwards, are found globally, including in regions where they are neither produced nor used directly[1]. Detection of trace amounts of PFCAs in complex environmental matrices presents analytical challenges[2]. In this project we show the application of an ultrasound-assisted extraction (UAE) requiring minimal solvent usage for extracting PFCAs from biosolids and soil. Samples from an agricultural setting were collected over a period of two years. The quantitative trace analysis of compounds was performed using LC-Orbitrap-MS. The setup allowed the analysis of PFBA (C_4) up to PFDoA (C_{12}) 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 28-minute gradient elution method. The MS detection in conjunction with LC was performed with an electrospay ionisation source (ESI). Results in biosolid-amended soil (N= 25) showed the presence of PFHpA (1.1 – 22.2 ng/g, N= 7), PFOA (2.7 ng/g, N= 1), PFNA (0.7 – 0.9 ng/g, N= 2), PFDA (0.1 – 1.8 ng/g, N= 7) and PFDoA (0.7 ng/g, N= 1). PFBA, PFPeA, PFHxA and PFUnA were not detected in amended soil samples. In contrast, control soil samples (N= 25) resulted in only three distinctive results above limit of detection, PFOA (1.3 ng/g, N=1) and PFDoA (0.5 – 23.2 ng/g, N= 2). Six batches of biosolids (N= 18) showed a wider range of concentrations between compounds, with PFBA (152 – 195 ng/g, N= 18), PFPeA (35 – 126 ng/g, N=18), PFHxA (31 – 74 ng/g, N= 18), PFHpA (6.8 – 24 ng/g, N=17), PFOA (4.5 – 9.9 ng/g, N= 18), PFNA (2.6 – 8.4 ng/g, N= 17), PFDA (2.8 – 10.1 ng/g, N= 17), PFUnA (0.3 – 3.5 ng/g, N=15) and PFDoA (6.9 – 10.5 ng/g, N= 18). Calibration ranges during this analysis varied between 10 – 500 ng/g and 0.1 – 500 ng/g depending on the compound and batch. The high PFCA content in biosolids can be identified as one route into an agricultural environment. However, not all PFCAs are retained in the analysed soil samples following this pathway making it essential to investigate the fate of PFCAs in the environment to a higher extend. On a global scale, especially in more remote regions, other pathways also need to be further investigated to explain ubiquitous detection in the environment.

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

2. Boxall ABA. New and emerging water pollutants arising from agriculture. *Organisation for Economic Co-operation and Development (OECD).* 2012.