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Evaluating micropollutant degradation and persistence in soil with compound-specific isotope analysis (CSIA)

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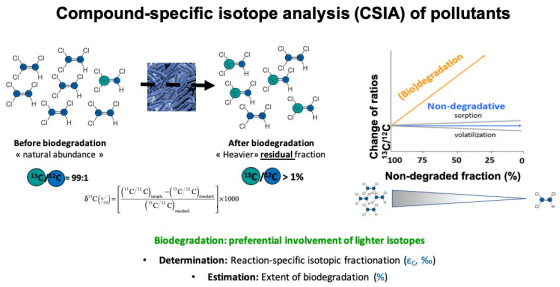
Knowledge on the occurrence, extent and pathways of (bio)degradation of micropollutants in soil is crucial to improve the monitoring of their persistence and predict ecotoxicological risks. Compound-specific isotope analysis (CSIA), based on natural abundance of stable isotopees, has successfully been applied to evaluate the transformation of industrial pollutants in groundwaters. However, the need of a sufficient mass of analyte for CSIA combined with low micropollutant, concentrations (sub-µg.g^{-1}) and the co-enrichment of non-volatile soil components, currently challenge micropollutant CSIA in soil [1]. Here, we show that QuEChERS are versatile preparation methods for precise and sensitive CSIA of micropollutants from soil samples, allowing adaptations for specific matrix-analyte combinations to reach more selective extraction. Micropollutants with contrasted physico-chemical properties, including pesticides and an antibiotic, were extracted from various types of soil from microcosms, mesocosms and field studies. No significant isotope fractionation for carbon ($\Delta\delta^{13}C \leq 1‰$) and nitrogen ($\Delta\delta^{15}N \leq 0.5‰$) was observed, despite variable extraction efficiencies. Column experiments with various soil types showed that biodegradation of anilide herbicides and fungicides mainly occurred in the soil solution of soil-plant systems, whereas degradation in soil remained limited [2]. In an agricultural catchment, we recently demonstrated the applicability of CSIA to track the degradation and export of the pre-emergence herbicide *S*-metolachlor from soil to surface water and identify the contributing source areas. Based on maximum change in carbon stable isotope composition ($\Delta\delta^{13}C = 4.6 \pm 0.5‰$) of *S*-metolachlor, we showed that degradation mostly (>95%) occurred in soil within two months after first application, leading to the formation of transformation products. In an urban context, CSIA of terbutryn [3], a biocide commonly added in facade paints and renders, highlighted its persistence in outdoor soil lysimeters and its potential transport into groundwater. Altogether, these studies emphasize the variability of micropollutant degradation in soil and proposes a framework using CSIA to examine the transformation of persistent and emerging micropollutant in polluted urban and agricultural soils.

[1] Elsner & Imfeld (2016), *Current Opinion in Biotechnology,* 41:60–72

[2] Pérez-Rodríguez et al., *Agriculture, Ecosystems and Environment,* 308:107257

[3] Junginger et al., *Chemosphere*, 305:135329



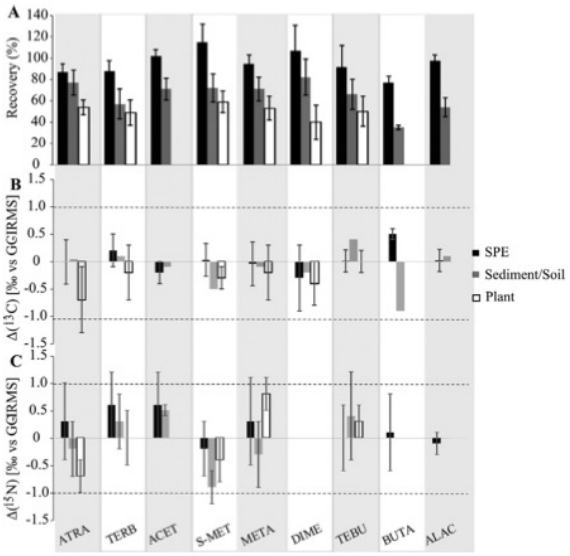


Figure 1. A - Extraction recovery, B – Effect on $\delta^{13}C$ ($\Delta(^{13}C)$ ‰ vs GC-IRMS), C – Effect on $\delta^{15}N$ ($\Delta(^{15}N)$ ‰ vs GC-IRMS) of SPE (water samples), and MUSE with DCM:Pentane (3:1) (sediment, soil, plant) for pesticides and all matrices combined. ATRA – atrazine, TERB – terbutryn, ACET – acetochlor, S-MET – S-metolachlor, META – metalaxyl, DIME – dimethomorph, TEBU – tebuconazole, BUTA – butachlor, ALAC – alachlor. Error bars denote standard deviation (1 σ, n ≥ 8). Dashed lines represent ± 1‰ (significance threshold) from reference isotope values of in-house standards measured with GC-IRMS.