

Development of methodologies for quantifying simultaneously PAHs and PCBs in sediment cores: advantages and drawbacks of chromatography coupled to thermal or solvent extraction and hyperspectral imaging.

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Polycyclic aromatic compounds (PAHs, PCBs) are widespread contaminants usually investigated in sediments, which act as sinks for these toxic persistent lipophilic compounds. Conventional approaches often use gas chromatography coupled to mass spectrometry (GC-MS) as a powerful tool to separate, identify and quantify them, but a preliminary step of extraction is required. The latter generally necessitate the use of toxic organic solvents and extend the duration of the overall analytical process, particularly for sediment cores analyses.

In this study, two extraction methodologies were optimized using experimental designs for multi-residual analysis and then compared: microwave assisted extraction (MAE) and thermal desorption (TD). Indeed solvent-free TD is frequently used for analysing PAHs from atmospheric particles but multi-residual analyses (PAHs/PCBs simultaneously) after TD from sediments are unusual. Several factors that may influence extraction recoveries were studied, including matrix parameters (sediment mass, organic matter content) and processing parameters (such as solvent volume for MAE). A full factorial design 2³ was used to optimise MAE extraction. Optimization of TD parameters was more complex as dozens of factors could be more or less influent, so a definitive screening design (DSD) was performed to screen the most influential factors (among 6 studied) and model the extraction recoveries at the same time.

After the optimization of extraction conditions, matrix effects were evaluated using the standard addition procedure and quality assurance and control (QA/QC) were implemented for comparing MAE and TD for sediment analysis. Although MAE process was easier to optimize and less sensitive to matrix effects, sediment thermal desorption could significantly improve the analytical process, due to direct coupling with TD-GC-MS/MS and complete automation. Moreover, it offered higher spatial resolution, particularly for sediment core analysis, due to the 1000-times lower sample size (mg instead g).

If TD-GC-MS/MS could significantly improve laboratory throughput for sediment analysis compared to MAE-GC-MS, a comparison was also carried out with hyperspectral imaging to determine the value of this methodology for sediment core analysis. Indeed, this recent, promising and non-destructive technology has the advantages of reducing drastically the analysis time (min instead of days) and has a considerably higher spatial resolution for sediment core analysis (µm instead of cm). But substantial work should continue to improve PAHs/PCBs signal identification and quantification, with a deeper understanding of matrix effects.