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Advantages of Matrix Solid Phase Dispersive (MSPD) Extraction Associated to MIPS versus MAE Liquid Extraction for the Simultaneous Analysis of PAHs, PCBs and Some Hydroxylated PAHs in Sediments

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Abstract : Sediments are complex environments which can accumulate a great variety of persistent toxic contaminants such as polychlorobiphenyles (PCBs), polycyclic aromatic hydrocarbons (PAHs) and some of their more toxic degradation metabolites such as hydroxylated PAHs (OH-PAHs). Owing to their composition, fine clayey sediments can be more difficult to extract than soils using conventional solvent extraction processes. So this study aimed to compare the potential of MSPD (matrix solid phase dispersive extraction) to extract PCBs, PAHs and OH-PAHs, in comparison with microwave assisted extraction (MAE). Methodologies: MAE extraction with various solvent mixtures was used to extract PCBs, PAHs and OH-PAHs from sediments in two runs, followed by two GC-MS analyses. MSPD consisted in crushing the dried sediment with dispersive agents, introducing the mixture in cartridges and eluting the target compounds with an appropriate volume of selected solvents. So MSPD combined with cartridges containing MIPs (molecularly imprinted polymers) designed for OH-PAHs was used to extract the three families of target compounds in only one run, followed by parallel analyses in GC-MS for PAHs/PCBs and HPLC-FLD for OH-PAHs. Results: MAE extraction was optimized to extract from clayey sediments, in two runs, PAHs/PCBs in one hand and OH-PAHs in the other hand. Indeed, the best conditions of extractions (mixtures of extracting solvents, temperature) were different if we consider the polarity and the thermodegradability of the different families of target contaminants: PAHs/PCBs were better extracted using an acetone/toluene 50/50 mixture at 130°C whereas OH-PAHs were better extracted using an acetonitrile/toluene 90/10 mixture at 100°C. Moreover, the two consecutive GC-MS analyses contributed to double the total analysis time. A matrix solid phase dispersive (MSPD) extraction procedure was also optimized, with the first objective of increasing the extraction recovery yields of PAHs and PCBs from fine-grained sediment. The crushing time (2-10 min), the nature of the dispersing agents added for purifying and increasing the extraction yields (Florisil, octadecylsilane, 3-chloropropyle, 4-benzylchloride), the nature and the volume of eluting solvents (methylene chloride, hexane, hexane/acetone…) were studied. It appeared that in the best conditions, MSPD was a better extraction method than MAE for PAHs and PCBs, with respectively, mean increases of 8.2% and 71%. This method was also faster, easier and less expensive. But the other advantage of MSPD was that it allowed to introduce easily, just after the first elution process of PAHs/PCBs, a step permitting the selective recovery of OH-PAHs. A cartridge containing MIPs designed for phenols was coupled to the cartridge containing the dispersed sediment, and various eluting solvents, different from those used for PAHs and PCBs, were tested to selectively concentrate and extract OH-PAHs. Thereafter OH-PAHs could be analyzed at the same time than PAHs and PCBs: the OH-PAH extract could be analyzed with HPLC-FLD, whereas the PAHs/PCBs extract was analyzed with GC-MS, adding only few minutes more to the total duration of the analytical process. Conclusion: MSPD associated to MIPs appeared to be an easy, fast and low expensive method, able to extract in one run a complex mixture of toxic apolar and more polar contaminants present in clayey fine-grained sediments, an environmental matrix which is generally difficult to analyze.

Keywords : contaminated fine-grained sediments, matrix solid phase dispersive extraction, microwave assisted extraction, molecularly imprinted polymers, multi-pollutant analysis
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