

BENEFITS OF A BINARY MODIFIER WITH BALANCED POLARITY FOR AN EFFICIENT SUPERCRITICAL FLUID EXTRACTION OF PAHs FROM SOLID SAMPLES, FOLLOWED BY HPLC

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ABSTRACT

Polycyclic aromatic hydrocarbons (PAHs) are widely distributed environmental contaminants. Their presence in soils indicates a possible contamination of ground water used for human purposes. Only with a rapid and efficient method could they be analysed in a routine setting.

Although there are a great number of publications on this subject, there are many differences between their ideal SFE conditions and related extraction recoveries.

With improvements on the extraction capacity of the supercritical fluid such as higher pressure, higher temperature and a

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stronger modifier effect, the developed method intends to be more robust and applicable to a larger range of matrices.

The optimal conditions consist on a 15 minutes extraction at 45 MPa pressure and 95°C. The supercritical fluid is a ternary mixture of CO₂ modified with methanol/dichloromethane 5:1. The separation and quantitation was performed by HPLC coupled with a diode array detector in series with a fluorescence detector, which combines high sensitivity with the possibility to confirm the components' identity in a complex chromatogram. Three of the six studied PAHs show recoveries above 90% and the others, those of higher molecular weight, around 70 – 90%.

This modified SFE procedure was applied to three environmental samples and fulfils the requirements of rapidity, high extraction efficiency, and simple performance.

INTRODUCTION

Polycyclic aromatic hydrocarbons mainly result from incomplete combustion of organic materials, in particular fossil fuels, and in smaller amounts, naturally by forest fires and possibly microbiological synthesis. Other specific sources are railroad bed soils and street dust. As a result, they are widespread in soil, air, and water.(1,2)

Analysis of micropollutants is one of the most important applications of supercritical fluid extraction, applied here to extract PAHs from soils, according to 80/778/EEC Directive. Their natural degradation is difficult and dependent on their molecular weight as well as on environmental conditions. Therefore, they are persistent compounds in the environment due to their chemical stability and biodegradation resistance, showing a tendency to accumulate in different environmental compartments.(2,4)

Regardless of several studies published about supercritical fluid extraction, its application to a specific sample, and a given analyte always had to be optimised to get its better profits.(8) Supercritical fluids have physical properties, such as low viscosity, high solute diffusive power, and density-linked solvent strength that make extraction selectivity and automation feasible, thus, offering a promising alternative to traditional extraction methods.(9) Using the conventional extraction methods, the analyst is often confronted with complex extracts requiring clean up and fractionation procedures to isolate the target analyte from all co-extracted compounds.

Reduced extraction time (fast sample preparation), mild extraction temperatures, high selectivity, reduced solvent consumption and waste, and, sometimes, less concentration and fewer sample clean up procedures are considered important advantages of supercritical fluids techniques.(7,10,11)