UNIVERSITI TEKNOLOGI MARA

ANALYSIS OF ORGANIC CONTAMINANTS IN WATER AND SOIL/SEDIMENT: METHOD DEVELOPMENT AND THE APPLICATION IN POLLUTION TRACKING

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ABSTRACT

Several organic contaminants (polycyclic aromatic hydrocarbons (PAHs), chlorpyrifos, sterols, phenol and pentachlorophenol (PCP)) were selected as possible chemical markers in pollution tracking. The extraction of these contaminants from water and sediment samples is the critical and time consuming step in the analysis as they are present in trace amounts. Thus, the focus of this study is to develop efficient, selective, and rapid analytical methods in determining these contaminants in water samples. Since the selected compounds are of wide range polarity, a tandem-cartridges solid phase extraction (SPE) using octadecyl bonded silica (C_{18}) and polymeric sorbent (LiChrolut EN) was found to be efficient with the advantage of minimising sample volume and reducing analysis time. Using this approach, the non-polar and moderately polar compounds were trapped in the C_{18} sorbent while the more polar compounds were trapped in the polymeric sorbent. Good recoveries of non-polar compounds, PAHs (81.8-92.7%) and chlorpyrifos (104.2%) were obtained from C_{18} cartridge using n-hexane in the first elution, while moderately polar compounds, sterols (89.7-92.9%) and PCP (73.6%) were recovered in the second elution using stronger eluting solvent (dichloromethane (DCM)). Acceptable recovery of phenol (82.9%) was achieved from LiChrolut EN cartridge using DCM as the eluting solvent. For soil/sediment samples, pressurised liquid extraction (PLE) was recognised as an efficient extraction technique. However, the use of high pressure and temperature resulted in dirty extracts and clean-up step is required prior to gas chromatography (GC) analysis. In this study, a clean-up step was done simultaneously with extraction by the addition of polar sorbent inside PLE extraction cell. Combination of polar sorbent and proper choice of solvents was able to establish an extraction with simultaneous clean-up for various organic contaminants from soil/sediment whereby polar and non-polar compounds were successfully separated. By incorporating silica into the extraction cell, PAHs, chlorpyrifos and pentachlorophenol were recovered in the first fraction using n-hexane, while phenol and sterols were recovered in the second fraction using methanol with good recoveries (PAHs from 87.1-96.2%, chlorpyrifos 102.9%, sterols from 93.7-100.5%, phenol 91.9% and pentachlorophenol 106.2%). Optimised PLE conditions were extraction temperature of 125 °C, extraction pressure of 1400 psi and 10 minutes static extraction time. The efficiency and precision of the developed PLE method were comparable with those of the existing EPA Method 3545. The developed SPE and PLE methods were applied to water and soil/sediment samples from point and non-point pollution sources. The data sets obtained were subjected to chemometric analysis including cluster analysis (CA), discriminant analysis (DA) and principal component analysis (PCA). Sterols was found to be strongly correlated to sewage and chicken farm samples while PAHs and pesticides were correlated to samples from agricultural activities. Analysis of water and soil/sediment samples from Langat and Klang River Basins suggested that the main sources of contamination in Langat River Basin were domestic sewage, agricultural activities and industrial activities/urban socioeconomic while the major sources of contamination in Klang River Basin were industrial activities, urban socioeconomic and domestic sewage, with minimal input of agricultural activities. These findings were in agreement with the inventory of local activities by the Department of Environment, Malaysia.

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CONTENTS