UTS UNIVERSITY OF TECHNOLOGY SYDNEY

Solvent-Assisted Headspace Sampling and Physical Investigation of SPME Fibres

By

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Certificate of Authorship and Originality

I certify that the work in this thesis has not previously been submitted for a degree, nor has it been submitted as part of the requirements for a degree except as fully acknowledged within the text.

I also certify that I have written the thesis, and any help that I have received in my research work and the preparation of the thesis itself has been acknowledged. In addition, I certify that all the information sources and literature used are indicated in the thesis. Also, “This research is supported by an Australian Government Research Training Program”.

Date: 15.01.2022

Rawaa Al-Baghdadi

DEDICATION

To my parent

And, I will always be grateful to my wonderful mother, who supported and surrounded me with her love, tenderness, and beautiful prayers that illuminated my path. If I have succeeded in my life, that is because you are in it.

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Abstract

The analysis of samples contaminated by organic compounds is an essential aspect of environmental monitoring. While pesticides are beneficial to crops, they have a harmful influence on the environment that must be considered when using them. Excessive use of pesticides may result in the extinction of biodiversity. Other human-related activities also lead to pollutants in the environment; these include polycyclic aromatic compounds (PAH) and aromatic (semi)volatiles. The term "semi-volatile" refers to a collection of contaminants having a wide range of chemical and structural characteristics. Analytes of interest in this study were PAH, Pesticide, TPH and discretionary aromatic volatiles. Because of the complex nature of environmental samples, isolating target organic compounds from their matrices is a significant challenge. In addition, trace organic components in water samples must be isolated and pre-concentrated to be analysed using analytical procedures. Therefore, sample preparation is a significant focus in environmental analysis nowadays. Over the past decade, the use of SPME in sample preparation has grown steadily. It is often combined with chromatographic separation modules to extract volatile and semi-volatile organic chemicals and allows the trace analysis of substances in complicated matrices.

In the present study, a solvent-free solid-phase microextraction (SPME) method has been developed to determine PAH, pesticides, TPH and discretionary aromatic volatiles in water and different matrices. The developed method was applied to various

environmental samples such as aqueous samples, milk, Orange juice and stream water.

The optimisation process will involve an analysis of the function of some commercially available fibres in achieving maximum analyte absorption and research of the various fibres' reactions to the solvents utilised, which has been investigated with each material. These fibres have also been subjected to solvent-assisted headspace analysis to determine their suitability. The method showed good linearity for 0.2 and 0.0005 $\mu g.mL^{-1}$ with regression coefficients ranging between 0.997 and 0.999. The relative standard deviation (RSD) (n = 6) for the target analytes were in the range of 4 –15 %, respectively. The developed technique was successfully applied to preconcentration and determination of the target analytes in environmental water and different matrices.

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DCM	Methylene chloride
GC-FID	Gas chromatography/flame ionisation detection
GC-MS	Gas chromatography/mass spectrometry
HSME	Headspace microextraction
ILs	Ionic liquids
LLE	Liquid-liquid extraction
LPME	Liquid phase micro-extraction
LOD	Limit of detection
LOQ	Limit of quantification
OCPs	Organochlorine pesticide
PDMS	Polydimethylsiloxane
PDMS/DVB	Polydimethylsiloxane / divinylbenzene
PDMS	Polydimethylsiloxane
PPM	Parts-per-million
POPs	Persistent organic pollutants
RS-HPLC	Reversed-phase high-performance liquid chromatography
RTIL	Room temperature ionic liquid
SDME	Single drop microextraction
SPE	Solid-phase extraction
SPME	Solid-phase micro-extraction
VOCs	Volatile organic compounds
PT-GC/MS	purge and trap (PT) gas chromatography-mass spectrometry
DSC	differential scanning calorimetry
TGA	Thermogravimetry

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