MASTER'S THESIS

Development of sample pretreatment methods for complex analytical matrices

Ho, Yee Man

Date of Award:
2013

Development of Sample Pretreatment Methods for Complex Analytical Matrices

HO Yee Man

A thesis submitted in partial fulfillment of the requirements

for the degree of

Master of Philosophy

Principal Supervisor: Dr. Kelvin S.-Y. Leung

The Hong Kong Baptist University

August 2013

Abstract

The determination of trace analytes in complex analytical matrices such as food and traditional Chinese medicines (TCMs), often requires an extensive sample preparation steps prior to instrumental analysis. These sample pretreatment steps include sampling, extraction, clean-up and pre-concentration. However, these steps are often time and solvent consuming, laborious and tedious. Recent evolution in sample pretreatment techniques focused on miniaturization in terms of sample size, solvent consumption and preparation time. More importantly, it aims to increase the extraction efficiency.

A diversity of research works in this thesis aim to facilitate the implementation of sample pretreatment methods for solid complex matrices such as food and TCMs. The innovations of the research works featuring a simplification with high-throughput sample preparation method, utilization of modified tools for extraction and a highly selective material with imprinted effects.

This thesis firstly describes a micro-scale extraction method, dispersive liquid-liquid microextraction (DLLME). A rapid, high throughput sample preparation method based on DLLME for simultaneous analysis of ten organophosphorus pesticides by gas chromatography-mass spectrometry (GC-MS) has accomplished a 100-fold enrichment with LOD down to sub-ppt levels based on 0.1g sample. The method has been fully validated in twelve commodities including fruits, vegetables and TCMs. A bi-functional ionic liquids (ILs) was used as a leachant and dispersive solvent in another DLLME study. An enrichment factor up to 450 was achieved by modifying a SPE cartridge pre-coated with ILs for multiple determinations of two different chemical groups, Sudan dyes and phthalate esters, from six oil-based paste and solid condiments. The pre-coated IL frits streamlined sample clean-up and extraction into a single step which shortens the extraction time into 6 min with low LOD in ppb levels. The third part of researches describe the design of molecularly imprinted polymer (MIP) in the selective extraction of cinnamic acid derived from TCMs. The strategic design involves a basic monomer, 4-vinylpyridine and cross-linker EGDMA. The MIPs showed a significant imprinting effect in application.

Table of Contents