

ABSTRACT

The use of chemical pesticides as crop protection agents is an indispensable feature of modern agriculture, which helps to ensure global food security. However, extended and indiscriminate use of these pesticides results in the accumulation of traces of these chemicals in the agricultural produce, termed as pesticide residues, which in turn cause harmful effects upon consumption of such produce. This makes pesticide residues a major food safety concern. Many countries across the world have issued increasingly stringent regulations of maximum residue limits (MRLs) for pesticide residues in various food commodities to ensure consumer protection. In this context, testing of pesticide residues in food is important to ensure compliance of food commodities with such regulations.

Spices are considered difficult matrices to analyse because of their complex chemical composition, with significant concentrations of active compounds that contribute to their special properties like colour, flavour and aroma. These compounds can potentially interfere with analysis of pesticide residues. Spices are also very diverse, and belong to different classes like dried fruits, seeds, floral parts, roots etc. which are distinct from one another. It is practically difficult to use a single analytical method to cover all major classes of spices. Thus, modern analytical methods for spices need to be sufficiently general to aid easy implementation but also have to be fine-tuned with respect to different classes of spices to ensure analytical performance. Development of such methods using UPLC-MS/MS and GC-MS/MS, covering different classes of spices, is documented in this thesis.

Part I of the thesis deals with developing, optimizing and validating pesticide residue analysis for different classes of spices. The pesticides most commonly used for cultivation of spices in India are covered. Two instrumentation techniques are used, viz. UPLC-MS/MS GC-MS/MS, with QuEChERS sample preparation method which was

optimized for different classes of spices. One of the most important problems faced in using mass spectrometric techniques for quantitative analysis is the matrix effect (ME), which makes response of a target analyte different in solvent and matrix extracts. This issue poses significant challenges in high sensitivity trace analysis for pesticide residues, especially in complex matrices like spices. Novel ways of addressing ME in pesticide residue analysis in spices is also documented in Part I.

Part II of the thesis deals with application of the methods developed in Part I to real samples for the purpose of evaluation of compliance with national MRLs as well as characterization of food safety hazards due to presence of pesticide residues in commonly consumed spices. A total of 60 market samples were analysed for 78 pesticides using UPLC-MS/MS and GC-MS/MS. Based on the results obtained, compliance with the national MRLs and food safety hazard characterization calculations were performed.