CHAPTER - IV
6.0 CONCLUSIONS

The proposed MSPD-GC-MS method can be readily applied to the extraction of thirteen pyrethroids in 0.5 g of goat milk and tissue sample (liver, muscle, heart and kindly). C18 material was used as MSPD dispersion adsorbent and MWCNTs were used as cleanup adsorbent. Twenty milliliters of n-hexane and acetone (7:3, v/v) was used as an elution solvent. This procedure described above is simple, rapid and the GC-EI-MS detection provides excellent selectivity and sensitivity for the quantitative analysis of those analytes in different tissue and milk samples that can content the requests of some directives.

A new method based on solid phase extraction (SPE) and high performance liquid chromatography with UV detection was developed for the determination of TAA in BAL fluid. The suitability of different types of commercially available SPE cartridges used for the extraction of TAA was determined. SPE using Oasis HLB sorbent had the best performance in terms of cleanup efficiency and achieved the highest recovery under the experimental conditions tested. The SPE-HPLC-UV method proved sensitive, selective, accurate, precise and efficient at recovering TAA from BAL fluid.

The proposed MSPD method can be readily applied to the extraction of azimsulfuron in 0.5g of milk and tissue sample (liver, kidney, muscle and heart). This analytical method is suitable for the quantification of azimsulfuron in milk and tissues (liver, kidney, muscle and fat) at levels down to 0.02 mg/kg (ppm). Average recoveries at the limit of quantification ranged from 87.6 to 104.8 % with standard deviations in the range of 2.62 to 6.96. Good repeatability was demonstrated for all matrices. The method was demonstrated to have no interferences from the matrix.

The developed GC-MS method for the analysis of dithiocarbmates in aquatic culture mediums provides fast and accurate results and has grater advantage over classical methods such UV-VIS and GC-FPD in terms of precise quantification of CS\textsubscript{2} over a wide range from 0.01 mg/L to 100 mg/L.
Rapid, simple, sensitive and eco-friendly extraction methods such as US-DLLME and F-HLLME combined with GC-MS were developed and validated for the determination of Fipronil and its metabolites (desulfinyl, sulfone, and sulfide) in human plasma, urine and soil samples. These simple sample preparation procedures represent major advantages, making this method suitable for determination of trace levels of both parent and its metabolites in a single run in combination with GC-MS. These methods showed comparable and acceptable recoveries in the range between 86.9-104.5% (RSD < 7.9%) from complex samples such as plasma, urine and soil.

A novel and efficient method for the extraction of pyrethorid residues in human plasma samples was developed using MSPE method coupled with GC-ECD. The pyrethroid residues in MSPE extracts were confirmed by GC-MS. The recoveries were obtained in the range of 89.7% to 104.3% and LOD and LOQ were 3 to 10 ng mL⁻¹ and 10 -30 ng mL⁻¹, respectively. Furthermore, a fast separation time (5min) was achieved for 5 mL of sample volume avoiding time-consuming, column-passing process of conventional solid phase extraction. The results indicated that the proposed method was suitable for the rapid qualitative and quantitative determination of pyrethroids in human plasma samples.

The proposed LC-MS method can be readily applied for the identification and quantification of A-KG in human plasma samples. Prior to LC-MS analysis, a simple derivatization method with methyl imidazole was successfully applied and imidazole derivative of A-KG was formed. The obtained derivative was detected with the characteristic fragments (m/z 179 and 197) with mass spectral analysis. Linearity was achieved over the concentration range of 100-2500 ng mL⁻¹. The validation data showed that the developed method is specific, precise, accurate, robust and rugged.

A rapid, sensitive, and eco-friendly multi-residue method based on a MSPD-UA-DLLME extraction procedure and HPLC-DAD, LC-MS/MS analysis, has been developed and validated for bovine milk and honey samples. The simple sample preparation procedure represents major advantages, making this method suitable for determination of five neonicotinoids in matrix rich samples such as, milk and honey.
A single drop microextraction procedure was developed for the analysis of phthalate esters in water, and experimental conditions affecting the extraction efficiency were optimized. The modification in the extraction vial has increased the rotation speed and enabled the efficient extraction of phthalate esters in water samples. This method is simple, fast and requires only small volume of organic extractant, which is suitable for the analysis of phthalates in water samples.

A simple, rapid and sensitive SPE-LC-MS/MS method has been successfully applied to the determination of pesticide residues (Metazachlor, Buprofezin and Cloquintocet-mexyl) in ADM patient’s blood samples. The method developed shows satisfactory validation parameters in terms of linearity, low limits, accuracy and precision. The average recoveries at the spiked levels are ranged between 92.1–105.3%. The uncertainty associated with the analytical method, expressed as RSD, was lower than 4.3% for all compounds tested.