CHAPTER 4

CONCLUSION

The proposed GC–FPD method and single drop microextraction conditions were found to be applicable to the determination of dimethoate and malathion in vegetable and fruit samples. GC–FPD conditions for the determination of dimethoate and malathion, including extraction using SDME technique, were investigated. The parameters affecting for the separation of dimethoate and malathion were optimized which included type of column, temperature of injector, temperature programmed, temperature of detector and flow rate. Investigation of the optimal SDME conditions consisted of type of extraction solvent, drop volume, stirring rate, salt concentration, sample pH and extraction time. Limits of detection, percentage recovery, intra and inter–day repeatability and linearity were also studied. Finally, these optimal conditions were used in determination of dimethoate and malathion in vegetable and fruit samples.

The optimum separation of dimethoate and malathion was obtained by appropriately adjusting the temperature of injector, oven temperature programmed, temperature of detector and flow rate. The injection port was held at and used in splitless mode. The oven temperature program was from 200ºC to 250ºC at the rate of 20ºC/min and held at 250ºC for 3 min. The flow of helium gas was 1 ml/min. The optimal conditions of other parameters, which affect the FPD operation, are summarized as follows: 200ºC of FPD temperature, 60 ml/min of make-up gas (N_2), 70 ml/min of hydrogen and 100 ml/min of air. This method could be used to separate

the dimethoate and malathion compounds, which gave sufficiently well shaped peaks (resolution > 1.5) in approximately 5 min.

The SDME was employed for the extraction of analytes. Toluene was used extraction solvent. The optimized parameters for the SDME process are as follows: 2.5 µl of drop volume, stirring rate at 100 rpm, addition of 20% of salt concentration (NaCl), pH at 6 and extraction time for 5 min. The accuracy expressed in term of percentage recovery of this method for dimethoate and malathion was found to be from 87.6 to 103.4%. Limits of detection were 10 µg/L for dimethoate and 5 µg/L for malathion. The precision expressed in term of intra and inter-day repeatability that exposed in term relative standard deviation. The intra-day repeatability was found between 1.4-4.0% for dimethoate and 1.6-3.7% for malathion. Inter-day repeatability for dimethoate and malathion was obtained in the range 1.1-2.7% and 0.5-2.0%, respectively. Linearity of all pesticides was obtained in the range 10–1000 µg/L.

The developed methods for simultaneous determination of dimethoate and malathion offer superior performance characteristics, i.e. a simple method, low cost, rapid and less amount of waste. These methods were applied to the determination of dimethoate and malathion in vegetable and fruit samples that had been collected from different places. Dimethoate was found in some samples, but malathion was not detected in all samples. The concentration of dimethoate obtained was in the range of 59–943 µg/L in some orange samples. These methods offer simple method, rapid and low cost for the determination of dimethoate and malathion in vegetable and fruit samples.

Suggestion of further work

In the case of SDME technique, the sample of SDME extraction technique should be quite clear other wise it is hard to see the solvent drop. Additionally, the microsyringe should be rinsed for several times because of the efficiency and effectiveness of the results.

THE RELEVANCY OF THE RESEARCH WORK TO THAILAND

Dimethoate and malathion are a group of organophosphorus pesticides (OPPs) which are widely used for control of pests, weeds and diseases in a wide variety of plantation products. Moreover, in many countries still use them because of their high affectivity and relatively low price. However, there is some bad point as well. For instance, the wastes that are released from manufacturing, transportation, or agriculture applications normally have these compounds (dimethoate and malathion) to be part of them, which are very toxic and can be absorbed by human organisms because of acetyl-cholinest estrase de-activation. Toxicity of them can lead to the possibility of death depending on absorption of the organisms. Therefore, it is essential to determine the amount of dimethoate and malathion in various vegetable and fruit samples. The several methods based on GC-FPD have been developed for determination of both. Due to the low concentrations of these compounds and complex matrices, vegetable and fruit samples are not directly analyzed using these approaches. Currently sample preparation is performed by single drop microextraction (SDME) widely used for determination of these compounds. Therefore, in this research work, SDME-GC-FPD methods were developed for routine analysis of these compounds in variety sample such as natural water, wastewater and pharmaceutical samples, which are rapid extraction, simple tool when compare with SPME technique.