CHAPTER 4

CONCLUSION

Firstly, a liquid chromatographic method has been developed for the qualification of methomyl, carbofuran and carboxin. The µBondapak C_{18} column was selected because of its wide versality for retention of most compounds and has a history of documented application usage. HPLC optimum condition was developed by employing 40% ACN/ H_2O of mobile phase at 0.8 ml/min detected by 233 nm UV detector.

A validation of the method was performed in terms of precision, linearity range, and limit of detection and limit of quantification of the method. All of the parameters were needed as a part of an evaluation of the instrument efficiency. Satisfactory reproducibility precision in terms of % RSD of the retention time and peak area was in a range of 0.61 to 2.83% and 0.02 to 0.67% respectively. The repeatability precision values in terms of % RSD of the retention time and peak area was in the range of 0.12 to 0.22% and 2.24 to 2.7%. Good linearity of the response was found for all pesticides with linear determination coefficients higher than 0.9962. The LOD values were in the range of 0.02 to 0.06 µg/ml. The LOQ values in the range of 0.08 to 0.18 µg/ml were obtained.

In the step of the SPE, C_{18} sorbent and florisil sorbent were compared at different level of concentrations to evaluate the efficiency of the SPE Sep-Pak. Higher % recoveries ranging 87.1% – 106.41% at 0.5, and 1.0 ppm fortification levels was obtained by using florisil sorbent. The characteristic of the analytes, concentration of

the components, characteristics and amount of the sample matrix affected to the retained capacity of the SPE. The optimum condition for SPE was achieved by conditioning with 10 ml of methanol followed by 10 ml of Milli Q water. It is necessary to make sure that the SPE cartridge should not be dried before loading the sample. It can affect the percent recovery of the method. The optimum eluent was 3 ml of 70% ACN/ H_2O.

Accuracy of the extraction method was calculated through the recovery of each pesticide. In the recovery studied, matrix effects occurred and the background peaks co-eluted with the interest of compounds. This matrix effects interfered the identification and quantitation of the analytes resulting low percent recovery. Especially polar pesticides methomyl can not be detected under the optimized condition. Therefore condition was further optimized. Proper detection wavelength was changed from 233 nm to 205 nm. Adequate separation was achieved by decreasing the mobile phase 40% ACN/H_2O to 25% of ACN/ H_2O. Adequate average percent recovery obtained was 94.68% and 90.03% for carbofuran and carboxin respectively.

In the extraction step prior to analysis, it was found that instead of using a blender, chopping the sample into small pieces was effective to minimize the matrix effects. In order to compensate the matrix effect, the amount and concentration of sample matrix in the loading step of SPE have to be strongly considered. Large amount and concentrated sample matrix affected the retained capacity of the SPE to an extent resulting in a matrix enhancement effects. Therefore, it was proposed that, smaller amount of extract to be used for the clean up. In addition, centrifugation was essentially help to settle down the solid matter before passing the SPE.

In this experiment, carbofuran residue was not detected in all cabbage samples resulted in no potential risk to consumers. The amounts of carboxin found in vegetables were in the range from 0.54 to 4.14 mg/kg. The residues in the sample with the safety label are likely to be at lower level than those in the sample without safety label. 20 % of analyzed sample with safety label and 33 % of analyzed sample without safety label gave positive values. Low residue in the cabbage with the safety label may be due to the unavoidable environmental contamination such as drift or irrigation water supplies or occasional mislabeling. For the human risk assessment, the highest concentration of the carboxin was compared with RfD value. If the cabbage was contaminated by 4.14 mg/kg of fresh weight, a normal Thai man weighing about 60 kg would consume 0.04 mg of carboxin, corresponding to 0.0006 mg/kg body weight/day and this amount is much lower than the 0.1 mg/kg/day of RfD value. There was no great potential risk for human health and an average man could take this contaminated cabbage without harmful effect to him.

In conclusion, extraction of carbofuran and carboxin using solid phase extraction combined with HPLC has been demonstrated as a reliable analytical tool for the residues analysis. Even though, methomyl pesticides could not detected in the sample extract, because of the broad spectrum use of methomyl in agriculture protection and toxicity effects to humans, more data about contamination in the soil, water and food stuff are necessary to monitor to assess the potential risk to human health. No residues of carbofuran and low level of carboxin residues were detected in the cabbage sample resulted in no potential risk for human health after cabbage consumption. It can be suggested that the consumers who seek to reduce their

exposure to pesticides can do by choosing food with the safety label. It is highly recommended for the consumers to decrease the amount of pesticides remaining as much as possible such as thoroughly washing vegetables. In addition, better producers and clear policies are needed to make ensure that foods essentially free of detectable residues. Alternative pesticides which are safety to humans and the environment, training about proper application of pesticides are also necessary. Finally, the future researchers are recommended to study about the pesticides residues for long term monitoring programs in enlarged areas and potential health risk of pesticides exposure to humans in Thailand.