CHAPTER 4

CONCLUSION

Canned fish are frequently and largely eaten in Thailand, so their toxic metal content should be of some concern to human health. Solder, used in the manufacture of cans, is a recognized source of contamination of food by lead during canning (Voegborlo *et al.*, 1999). The concentration of lead in canned fish samples are usually found at the trace levels. Stripping voltammetry techniques, particularly anodic stripping voltammetry (ASV) and adsorptive cathodic stripping voltammetry (AdCSV), is a powerful analytical technique for the determination of ppb levels of metal ions (Shemirani1 *et al.*, 2005).

The first part of this work was the study of chemically modified carbon paste electrodes with group of xanthone compounds (xanthone, xanthene, thioxanthone and acridone) for the determination of Cu(II), Cd(II), Hg(II) and Pb(II) by differential pulse anodic stripping voltammetry. Two different modified carbon paste electrodes (7.5% and 15% w/w) were tested for their voltammetric signals. The standards solution were prepared at the concentration of 5 mg L⁻¹ Cd(II) in 0.3 M CH₃COONH₄, 5 mg L⁻¹ Cu(II) in 0.2 M acetate buffer, 10 mg L⁻¹ Hg(II) in 0.2 M acetate buffer and 5 mg L⁻¹ Pb(II) in 0.2 M HNO₃. The result shown that group of xanthone compounds, without additional function group, is not suitable for quantitative analysis of these metals at the condition under study.

The second part of this work was the study of adsorptive cathodic stripping voltammetric method for determination Pb(II). The method is based on the adsorptive accumulation of 8-hydroxyquinoline complexes of Pb(II) onto a hanging mercury drop electrode, followed by reduction of adsorbed species by voltammetric scan using square wave pulse modulation. The optimum experimental conditions and parameters were found to be 0.1 M CH_3COONH_4 as the supporting electrolyte, pH of 7.5, a 8-hydroxyquinoline concentration of 15 μ M, accumulation potential at -0.7 V (vs. Ag/AgCl) accumulation time of 120 s, scan rate of 0.3 V/s and pulse amplitude of 20 mV.

At the optimized conditions, the linear calibration graph was obtained in the concentration range 0.5 - 90.0 μ g L⁻¹ with correlation coefficient 0.9973, the limit of detection (LOD) is 0.108 μ g L⁻¹ and the limit of quantification (LOQ) is 0.360 μ g L⁻¹.

The effect of interference from matrices was studied in canned fish, the result showed that the slope of standard addition and calibration were not parallel. Therefore standard addition curve was used for Pb(II) determination in canned fish.

The accuracy of this technique was evaluated from the percent recovery. The recovery values were obtained in the range 93.68 - 95.13%. The relative standard deviation (n = 10) at lead concentrations of 1.0, 5.0 and 10.0 μ g L⁻¹ were 6.23%, 2.40% and 2.00% respectively. Possible interferences by metal ions, which are of great significance in real matrices, have been studied.

For the quantitative analysis of lead in canned fish samples. The concentration of Pb(II) in canned fish samples (wet weight) were found in range $0.121 - 0.285 \ \mu g \ g^{-1}$. However, the concentration of Pb(II) in canned fish samples were lower than the food contamination standard limited level (< 1.00 $\mu g \ g^{-1}$) issued by the Ministry of Public Health of Thailand.

The adsorptive cathodic stripping voltammetry method in this research was successfully due to the method is simple, sensitive, inexpensive and rapid for the determination of Pb(II) and could be applied to determination of Pb(II) in canned fish.