CHAPTER 4

CONCLUSIONS AND SUGGESTION FOR FURTHER WORK

4.1 Conclusions

This research work consists of two parts. The first part, a low cost FI spectrophotometric method for the determination of cadmium(II) has been proposed. It has been possible to construct simple flow injection systems for the determination of trace cadmium(II) at the mg L^{-1} level. Flow Injection analysis (FIA) systems were developed and constructed from easily available materials and instruments. The method presented good reproducibility, high sensitivity, and sample through put. The method was also simple, inexpensive and reliable. This method was successfully applied to the analysis of wastewater samples from various districts in Chiang Mai Province. The second part, SIA with spectrophotometric detection procedure was developed for cadmium(II) determination. SIA offers precise automatic control of reaction conditions and microfluidic manipulation of samples and reagents. It also operated at microliter scale, saving reagents while generating small volumes of waste.

4.1.1 FIA spectrophotometric determination of cadmium(II) using rhodamine B as A complexing agent

A flow injection spectrophotometric procedure for cadmium(II) determination based on complexation of cadmium(II) with KI and rhodamine B has been developed in which a reagent solution or standard solution was injected into the sample or standard solution stream, The complex was formed at pH 3.0. The absorption of which was then measured at 612 nm. Optimum conditions for determining cadmium(II) were investigated. Several factors influencing the sensitivity of the method were optimized using the univariate method and the optimum conditions are summarized in Table 3.12. A linear calibration graph over the ranges of 0.00-1.00 mg L^{-1} with a slope could be established with a regression equation: $y = 0.824x-0.0137$ with the correlation coefficient of 0.9996. The method was very sensitive as detection limit (LOD) as 0.007 mg L^{-1} cadmium(II) could be determined. The relative standard deviation (%RSD) for determining 0.2 mg L^{-1} of cadmium(II) was 1.08% (n=11), and the sample throughput of 60 h^{-1} were obtained. High accuracy with percentage recovery value of spiked wastewater sample was found to be 101.31% for cadmium(II). The proposed FIA method is free from interferences most common ions such as sodium, potassium, magnesium, calcium, and etc. The proposed FI spectrophotometric method for cadmium(II) has been carried out under optimum conditions (Table 3.12). The proposed FIA method has been applied to the determination of cadmium(II) in wastewater samples collected from different locations in Chiang Mai Province. The concentration of cadmium(II) were found to be in the range 0.015-0.069 mg L^{-1} which were in good agreement with the results obtained by ICP-MS method verified by *t*-test at the 95% confidence level.

The proposed FIA method is simple, rapid, inexpensive, accurate and reproducible which is suitable for the monitoring of cadmium(II) in wastewater samples.

4.1.2 SIA spectrophotometric determination of cadmium(II) using rhodamine B as A complexing agent

The sequential injection analysis (SIA) procedure for cadmium(II) determination based on complex with KI and rhodamine B has been developed, the resulting of complex Cd(II)-Iodide-Rhodamine B was measured at 612 nm. Optimum conditions for determining cadmium(II) were investigated by univariate method and the optimum conditions are summarized in Table 3.34. A linear calibration graph was over the ranges of 0.00-1.00 mg L^{-1} with a regression equation: y = 1.3968x+0.0146 with the correlation coefficient of 0.9997. The method was sensitive as detection limit (LOD) as 0.005 mg L^{-1} cadmium(II) could be determined. The relative standard deviation (%RSD) for determining 0.2 mg L^{-1} of cadmium(II) was 0.16% (n=11), and the sample throughput of 50 h^{-1} were obtained. High accuracy with percentage recovery value of spiked wastewater sample was found to be 100.54% for cadmium(II). The proposed SIA method is free of interferences from most common ions such as sodium, potassium, magnesium, calcium, and etc. The proposed SI spectrophotometric method for cadmium(II) has been carried out under optimum conditions (Table 3.34). The proposed SIA method has been applied to the determination of cadmium(II) in wastewater samples collected from different locations in Chiang Mai Province. The concentration of cadmium(II) were found to be in the range 0.015-0.065 mg L^{-1} which were in good agreement with the results obtained by ICP-MS method verified by *t*-test at the 95% confidence level.

The proposed SIA method is simple, rapid, inexpensive, accurate and reproducible which is suitable for the monitoring of cadmium(II) in wastewater samples.

4.1.3 Comparison of the analytical characteristics between FIA and SIA method for cadmium(II) determination

Comparison of the analytical characteristics between flow injection (FI) and sequential injection (SI) analysis summarized in Table 4.1. The results were obtained by FIA method offers a better analytical performance in terms of sensitivity and sample throughput (60 h^{-1}). The SIA system is more sensitive with a lower detection limit (0.005 mg L^{-1}) than the FIA system which has a detection limit of 0.007 mg L^{-1}. The reproducibility (R.S.D) of FIA and SIA methods are 1.08% (Intermediate level) and 0.16% (high level), respectively. The %recovery of FIA and SIA methods are 101.31% and 100.54%, respectively, which have high percentage recoveries of both methods. Nevertheless, the SIA method is the lower consumption of a reagent than the FIA system; it uses microlitres of the reagent and solution per analytical cycle, with minimum waste production.

Table 4.1 Comparison of the analytical characteristics between FIA and SIA method

Parameters	FIA	SIA
Linear range	0.00-1.00 mg L^{-1}	0.00-1.00 mg L^{-1}
Correlation coefficient (R^2)	$R^2 = 0.9996$	$R^2 = 0.9997$
Relative standard deviation	1.08 %	0.16%
Limit of detection	0.007 mg L^{-1}	0.005 mg L^{-1}
Limit of quantification	0.013 mgL^{-1}	0.010 mgL^{-1}
%Recovery	101.31%	100.54%
Sampling rate	60 h^{-1}	50 h^{-1}

4.2 Suggestion for further work

4.2.1 The proposed FIA and SIA manifolds can be improved for continuous monitoring. A flow injection system can be equipped with a computer or timer to automatically control the measurement and then evaluate data. An autosampler device can be used to obtain accurate and reproducible data.

4.2.2 The FIA and SIA systems should be standardized using standard cadmium(II) solution daily to ensure that reproducible results are obtained

4.2.3 With slight modification the FIA and SIA manifolds could be used to determine a wide range of analytes

4.2.4 An on-line preconcentration and separation by the FIA and SIA systems with a microcolumn can be used to make adequate concentration for instrumental used in case of samples containing trace amounts of cadmium(II)