

## **CHAPTER 4**

### **CONCLUSIONS AND SUGGESTION FOR FURTHER WORK**

#### **4.1 Conclusions**

This research work consists of two parts. The first part, a low cost spectrophotometric FIA method for the determination of iron (III) has been proposed. 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 bottled-drinking water samples available in markets in Chiang Mai Province. The second part, SIA with spectrophotometric detection procedure was developed for iron (III) determination. SIA offers precise automatic control of reaction conditions and microfluidic manipulation of samples and reagents. It also operated at microlitre scale, saving reagents while generating small volumes of waste.

#### **4.1.1 FIA spectrophotometric Determination of Iron (III) Using Eriochrome Cyanine R and Cetyltrimethyl Ammonium Bromide as A Complexing Agent**

A flow injection spectrophotometric procedure for iron (III) determination based on complexation of Fe (III) with ECR and CTMAB has been developed in which a sample or standard solution was injected into the reagents stream, yielding a ternary complex of Fe-ECR-CTMAB with the stoichiometry for 1:2:4. The complex was formed at pH 4.5-5. The absorption of which was then measured at 610 nm. Optimum conditions for determining iron (III) were investigated. Several factors

influencing the sensitivity of the method were optimized using the univariate method and the optimum conditions are summarized in Table 3.10. The two linear calibration graphs over the ranges of 0.01-0.35 mg L<sup>-1</sup> and 0.50–0.80 mg L<sup>-1</sup> with different slopes could be established with a regression equation:  $y = 0.5385x + 0.0056$  for 0.01-0.35 mg L<sup>-1</sup> iron (III) with the correlation coefficient of 0.9994 and  $y = 0.403x + 0.0759$  for 0.50–0.80 mg L<sup>-1</sup> iron(III) with the correlation coefficient of 0.9986 respectively. The method was very sensitive as little as 0.005 mg L<sup>-1</sup> iron (III) could be determined. The Relative standard deviation of 1.19% (n=11) for 0.1 mg L<sup>-1</sup> iron (III) and the sample throughput of 144 h<sup>-1</sup> were obtained. The interference effects have been examined and it has been determined that only Al (III) have to be masked by 0.0006 mol L<sup>-1</sup> of sodium fluoride. The proposed method has been applied to the determination of bottled-drinking water samples. It was seen that results obtained by the recommended method were good agreement with those obtained by ICP-MS verified by *t*-test. The proposed method is sample inexpensive and fast which is suitable for the monitoring of Fe (III) in drinking water.

#### **4.1.2 SIA spectrophotometric Determination of Iron (III) Using Eriochrome Cyanine R and Cetyltrimethyl Ammonium Bromide as A Complexing Agent**

The sequential injection analysis procedure for Fe (III) determination base on complex with ECR and CTMAB has been developed, the resulting of complex Fe (III)-ECR-CTMAB was measured at 610 nm. Optimum conditions for determining Fe (III) were investigated. The twelve parameters namely : pH, concentration of reagents, aspiration volume of reagents, flow rate, holding coil length, holding time and reaction coil were optimized by univariate method and the optimum conditions

are summarized in Table 3.33. The two linear calibration graphs were over the ranges of 0.02-0.45 mg.L<sup>-1</sup> and 0.45-1.0 mg L<sup>-1</sup> with different slopes could be established with a regression equation:  $y = 0.2325x + 0.0022$  for 0.02-0.45 mg L<sup>-1</sup> Fe(III) with the correlation coefficient of 0.9995 and  $y = 0.1210x + 0.0632$  for 0.45-1.0 mg L<sup>-1</sup> Fe(III) with the correlation coefficient of 0.9948 respectively. The method was sensitive as little as 0.012 mg L<sup>-1</sup> Fe (III) could be determined. The relative standard deviation of 2.17, 2.25, 0.85 and 0.38 % (n=11) for 0.04, 0.1, 0.5 and 1.0 mg L<sup>-1</sup> Fe (III) respectively and the sample throughput of 39 h<sup>-1</sup> were obtained. The interference effects have been examined and it has been determined that only Al (III) have to be masked by 0.0006 mol L<sup>-1</sup> of sodium fluoride. The proposed method has been applied to the determination of iron (III) in bottled-drinking water samples as mentioned above. It was seen that results obtained by the recommended method were in good agreement with those obtained by ICP-MS method verified by *t*-test. The proposed SIA method is simple and reliable for the determination of iron (III) in drinking water samples.

#### **4.1.3 Comparison of the analytical characteristics between FIA and SIA method for iron determination**

A comparison of the analytical characteristics between flow injection analysis and sequential injection analysis summarized in Table 4.1. The results obtained by flow injection method offers a better analytical performance in terms of sensitivity and sample throughput (144 h<sup>-1</sup>). The FIA system is more sensitive with a lower detection limit (0.005 mg L<sup>-1</sup>) than the SIA system. The reproducibility (R.S.D.) of both methods is of almost the same level. 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	0.01-0.35 mg L <sup>-1</sup> and 0.50-0.80 mg L <sup>-1</sup>	0.02-0.45 mg L <sup>-1</sup> and 0.45-1.0 mg L <sup>-1</sup>
Correlation coefficient	R <sup>2</sup> = 0.9994 and R <sup>2</sup> = 0.9986	R <sup>2</sup> = 0.9995 and R <sup>2</sup> = 0.9948
Slope	0.538 L mg <sup>-1</sup> and 0.403 L mg <sup>-1</sup>	0.2325 L mg <sup>-1</sup> and 0.1210 L mg <sup>-1</sup>
Relative standard deviation	1.19 %	2.25%
Limit of detection	0.005 mg L <sup>-1</sup>	0.012 mg L <sup>-1</sup>
Sampling rate	144 h <sup>-1</sup>	39 h <sup>-1</sup>

#### 4.2 Suggestion for further work

4.2.1 This developed system should be used for routine determination of iron (III) in many sources such as food samples, pharmaceutical formulations and biological material.

4.2.2 In order to improve the sensitivity, the effect of other cationic surfactants such as cetylpyridinium chloride, polyethoxylated tallow amine and benzalkonium chloride should be investigated for determination of iron (III).



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