CHAPTER IV
CONCLUSIONS

4.1 Conclusions

In this research work, consists of two parts. The first part was development of FIA-spectrophotometric methods for selenium. It has been possible to construct simple flow injection systems for the determination of trace selenium at the μg mL^{-1} level. Flow Injection analysis (FIA) systems were developed and constructed from easily available materials and instruments. The second was development of SIA-spectrophotometric method. SIA was known as second generation of FIA method in which its instrumentation tend to require small portable, fast and easy-to-use devices with cost effectiveness and low waste production.

4.1.1 Determination of selenium in water samples by FIA-spectrophotometry

A flow injection spectrophotometric procedure for selenium determination based on an oxidation of iodide with selenite producing iodine which formed complex with VB resulting in purple colored reagent which was then measured at 546 nm. Optimum conditions for determining Se(IV) were investigated. Various factors influencing the sensitivity of the method were optimized using the univariate method. The optimum conditions are summarized in Table 3.11. The linear calibration graph over the range of 0.05-2.50 mg L^{-1} was established (Figure 3.15) with a regression equation: $y = 0.0158x + 0.0044$ for 0.05-2.50 mg L^{-1} Se(IV) with the correlation coefficient of 0.9993. The method was very sensitive as little as 0.008 mg L^{-1} Se(IV) could be

determined. The reproducibility obtainable for determining 0.20 mg L^{-1} of selenium standard by the proposed method (n=11) was 0.31 %. The methods have been satisfactorily applied to the assay of selenium in water samples with the sample throughput of 52 h^{-1}. Validation of the proposed method for selenium determination was also performed by comparison of the results obtained by both the proposed and the standard method (ICP-MS) using the same samples. The method is simple, inexpensive, accurate and reproducible which is suitable for the monitoring of selenium(IV) in the water samples.

4.1.2 Determination of selenium in water samples by SIA-spectrophotometry

The SIA spectrophotometric instrumentation was adapted and used as the basis for the development of a simple, rapid and low waste, sequential injection procedure for determination of selenium using Variamine Blue as colorimetric reagent. The SIA instrumentation set up and the SIA signals were shown in Figures 2.2, 2.3 and 3.28, respectively. Conditions necessary for selenium determination by SIA method were established by univariate method (see Table 3.32). The linear calibration plot was obtained over the concentration range of 0.01-2.50 mg L^{-1} (Figure 3.29) with the regression equation $y = 0.0807x + 0.0023$ (r^2=0.9990). The method was found to be very sensitive with the LOD and LOQ of 0.0005 and 0.0015 mg L^{-1}. The repeatabilities obtainable for determining 0.05, 0.10, 0.60 and 2.00 mg L^{-1} of selenium standard by the proposed method (n=11) with in the sample day and after week were not greater than 0.27%, 0.40%, 0.33% and 0.39% respectively. The methods have been applied to the assay of selenium in water samples with the sample throughput of 68 h^{-1}. Validation of the proposed method for selenium determination was also

performed by comparison of the results obtained by both the proposed and the standard method (ICP-MS). It was found that results obtained by both methods were in good agreement. With suitable modification the SIA instrumentation can be used as a basis to the development of greener analytical methods for analyzing a wide range of real samples environmental such as pharmaceutical, agricultural samples etc.

Table 4.1 Comparison between FIA and SIA procedure for selenium determination

Parameters	FIA	SIA
Linear rang (mg L^{-1})	0.05 – 2.50	0.01 – 2.50
LOD (mg L^{-1})	0.008	0.0005
LOQ (mg L^{-1})	0.0253	0.0015
Sample throughput (sample h^{-1})	52	68
Volume of sample (ml h^{-1})	7	2
Volume of reagent (ml h^{-1})	25	2

4.2 Suggestion for further work

With respect to the flow injection and sequential injection system developed in this research (Section 3.1 and 3.2). It has been successfully applied to the determination of selenium in water samples. With slight modification, the system can be used to determination of a wide range of analytes based on selective chemical reactions. The developed FIA and SIA system offer the advantages of robot, extreme simplicity and rapidity, low cost, with minimum reagent consumption and waste generation. These advantages ensure good analytical characteristics and performance of the system. With further improvement of the FIA and SIA instrumentation system, the system will be as efficient as the routine method for determining not only metal in water samples but also a wide range of analytes in various sample matrices in the near future.