CHAPTER IV

CONCLUSIONS

A simple and cost effective GrFFF system was developed. Such a system could be assembled using components which are easily available (see Section 3.1.1). The performance of GrFFF was tested (Section 3.1.3) by using silica samples, one was silica gel broad size ranges (<10 μm and 10-20 μm), the other was fairly monodisperse chromatographic silica (5 and 10 μm).

Various experimental conditions, namely carrier flow rate, relaxation time and particle loading, were investigated using the samples with broad size ranges (<10 and 10-20 μm). The higher flow rates provide higher lift forces which elevate the particle further away from the accumulation wall so that they experience the higher velecity flow streams and thus were observed to elute at a shorter retention volume. When the relaxation time was increased, some changes in the retention time were observed. Using the 5 μm chromatographic silica particles, it was found that the sample loading should not be more than 40 μg for the run conditions used (see Section 3.1.4).

FIA-CL was developed by making use of a commercial liquid scintillation counter (LSC) as a chemiluminescence (CL) detector. The CL signal was produced by the catalytic oxidation of luminol. The rFIA-CL manifold for trace Fe determination was investigated for some parameters, namely the length of the mixing coil, hydrogen peroxide and luminol concentrations and the buffer systems with pH values. A mixing coil of 30 cm, 10^{-3} M H_2O_2, 8×10^{-6} M luminol and 0.01 M Na_2HPO_4-NaOH buffer at pH 11 were found to be suitable (Section 3.2.2).

GrFFF combined with rFIA-CL (Section 3.3.5) and ETAAS (both off-line and on-line) (Sections 3.3.3 and 3.4.2) was used for size-based Fe speciation. Model particles of silica (5 and 10 μm) were coated with hydrous Fe oxide (goethite). It was found that the rFIA-CL detected only a small proportion of the total Fe in the sample particles. In the case of the 10 μm and 5 μm particles, this is only about 1% and 5%, respectively of the total Fe obtained after aqua regia digestion. The average thickness of the goethite layer was calculated to be 5 nm (Section 3.3.4).

The GrFFF-ETAAS technique was also used to study several clay samples (Section 3.4.7). Calibration of particle size could be made using chromatographic silica particles (5 μm and 10 μm). Evaluation of the effectiveness of the slurry ETAAS analysis was carried out. The Fe content of the particles increased as particle size decreased. This was particularly prominent for particles <5 μm in diameter and may be due to an increase in the Fe coating density on the surfaces of the particles or higher amounts of Fe rich minerals in the smaller particles.

Recommendations of Further Investigation

The GrFFF used in this work could be combined with ICP-MS for multi-elemental analyses of particles. ICP-MS has been hyphenated with some other FFF but not yet with GrFFF.

GrFFF is only useful for micron size particle separations, therefore if submicron size particles between 0.05 to 2 μm or 0.001 to 0.1 μm are of interest, they should be investigated using sedimentation FFF or flow FFF, respectively. However, those FFF techniques require much more complicated instrumentation than GrFFF.