CHAPTER 6

EXPERIMENTAL

6.1 Instrumentations

Experiments were performed using bare fused-silica capillary (Polymicro Technology, Phoenix, AZ, USA), 45 cm length with 75 μm i.d. and a distance of 36.5 cm from the point of injection to the detection window. The analyses were performed on a 3D capillary electrophoresis system (Agilent HP, Waldbronn, Germany). Indirect UV detection was selected at 206 nm. A voltage of +25 kV and constant temperature of 25^{o}C were applied for the separation. The atomic absorption spectrophotometer (VARIAN Spectra 800) was used for the determination of cationic nutrients which were comparable with the results from the capillary electrophoresis system.

6.2 Chemicals

Analytical grades of K^+, Na^+, Ca^{2+}, Mg^{2+}, alanine (Merck, Darmstadt, Germany), NH_4^+ (Ajax, Finechem, Australia), Mn^{2+} (Carlo Erba, Milan, Italy), Imidazole (Fluka, Steinheim, Switzerland), 18-crown-6 ether (Sigma-Aldrich Steinheim, Germany) and atomic absorption standard solutions of Co^{2+} (1020 mg L^{-1}, Aldrich Chemical Co. Inc., Milwaukee, WI, USA) and Cd^{2+} (1007 mg L^{-1}, Sigma-Aldrich, Steinheim, Germany) were used. Solid fertilizer (N-P-K = 18-4-6, N-P-K = 16-8-8) and fluid fertilizer (N-P-K = 7-10-10, Ca-Mg, Mg and Mn) samples were bought from markets.

6.3 Procedures

6.3.1 Preparation of stock standard solutions 1,000 mg L^{-1}

Stock solution of potassium (K^+) was prepared by dissolving 0.0571 g of K_2SO_4 in 0.5% HNO_3 and make volume to 25 ml in volumetric flask.

Stock solution of ammonium (NH^+_4) was prepared by dissolving 0.0743 g of NH_4Cl in deionized water and make volume to 25 ml in volumetric flask.

Stock solution of sodium (Na^+) was prepared by dissolving 0.0636 g of NaCl in deionized water and make volume to 25 ml in volumetric flask.

Stock solution of manganese (Mn^{2+}) was prepared by dissolving 0.0782 g of $MnSO_4$ in 1%H_2SO_4 and make volume to 25 ml in volumetric flask.

6.3.2 Preparation of background electrolyte

The electrolyte was prepared by dissolving 0.0082 g of imidazole, 0.0079 g of 18-crown-6 ether and 0.0134 g of alanine in deionized water. Then, the solution was adjusted to pH 6 with acetic acid before transfer to a 10 ml volumetric flask and make up to the mark with deionized water. The solution was filtered through a 0.45 μm membrane before analysis.

6.3.3 Sample preparation

Solid fertilizer samples were prepared by ground, sieved and weighted. 10 ml of 0.5% HNO_3 was added and sonicated for 20 min. Then, the solution was transferred to a 25 ml volumetric flask and make up to the mark with deionized water. In case of fluid fertilizer samples were prepared by dilution in deionized water and make up to the mark in a 25 ml volumetric flask. All sample solutions were filtered through a 0.45 μm membrane and made dilution before analysis.