

CHAPTER III

EXPERIMENTAL SECTION

3.1 Materials

Poly (diallyldimethyl ammonium chloride) -- PDADMAC, QUAT or MERQUAT[®] having an average molecular weight of approximately 240,000 daltons, was supplied by Cargon Corporation (Pittsburgh, PA). Poly (diallyldimethyl ammonium chloride) monomer have the empirical formula $(H_2C=CHCH_2)_2N(CH_3)_2Cl$. The polyelectrolyte was purified using a 10,000 MWCO, spiral wound membrane in order to remove the lower molecular weight chains to the point only trace amounts of polyelectrolyte was detected in the permeate. Sodium chromate was analytical grade supplied by Reidel-deHaen with a purity of 99%. Sodium sulphate (AR grade) and sodium nitrate with a purity of 99.5% were obtained from Carlo Erba (Via G, Milano). *p* - Hydroxybenzoic acid (99%) and sym - diphenylcarbazide were manufactured by Fluka (Buchs, Switzerland). Ethyl alcohol absolute (99.8%) was obtained from Merck Company. Univar Company (Auburn, N.S.W) supplied potassium hydroxide (AR grade). Gracial acetic acid (99.9%) was obtained from J.T. Baker (Phillipsburg, NJ). Silver nitrate (AR grade) supplied from Merck Company (Darmstadt, Germany) was used to determined chloride ion. Deionized and distilled water was used to prepare all solutions in all experiment.

Spectrum[™] cellulose acetate (type C) ultrafiltration membranes from Spectrum Medical Company (Houston, Texas) were used in these experiments with molecular weight cut-off equal to 10,000 daltons.

3.2 Analysis Methods

3.2.1 Determination of Chromate Ion

The concentrations of chromate in permeate and retentate solutions were colorimetrically determined by UV/VIS spectrophotometer. Sym - diphenylcarbazine was added to the solutions to form complexes with chromate ion. Then the absorption of solution was determined with a UV/VIS spectrophotometer (Perkin Elmer, Lambda 16) at 541.2 nm.

Sym - diphenylcarbazine reagent was prepared by dissolving 0.1 g sym - diphenylcarbazine in 50 ml ethyl alcohol and adjusted the volume to 250 ml by adding the solution of 10% acetic acid in distilled water.

3.2.2 Determination of Sulphate and Nitrate Ions

The concentrations of sulphate and nitrate in permeate and retentate solutions were determined by ion chromatography (Hewlett Packard Series 1050) with Altech Anion/R column (1 μ m, 150 mmx4.6 mm) and conductivity detector (Alltech 350). The sample solution was loaded into 20 μ l sample loop and injected into the column using 5 mM *p* - hydroxybenzoic acid as a mobile phase at the flow rate equal to 1.0 ml/min. Retention time of sulphate and nitrate were 10.7 and 7.0 minutes, respectively.

5 mM *p* - hydroxybenzoic acid was prepared by dissolving 0.6906 g *p* - hydroxybenzoic acid in distilled water, adjusted to pH = 8.5 by potassium hydroxide and made the volume to 1000 ml with distilled water.

3.2.3 Determination of Total Organic Carbon

The concentration of QUAT in the permeate and retentate solutions were determined by a total organic carbon (TOC) analyzer (TOC-5000A, Shimadzu) as described by the following procedures:

3.2.3.1 Measurement of TC (Total Carbon)

A TC combustion tube is filled with oxidation catalyst and heated to 680°C. Carrier gas (high purity air) is supplied into this tube after it is controlled at flow rate of 150 ml/min by a pressure controller and a mass controller and moistened by a humidifier. When sample 100 µl has been introduced by a sample injector into the TC combustion tube, TC component in the sample combusted or decomposed to become CO₂. The carrier gas which contains combustion product from the TC combustion tube flows through an IC reaction vessel cooled and dried by a dehumidifier. It is then sent through a halogen scrubber into a sample cell set in a non-dispersive infrared gas analyzer (NDIR) where CO₂ is detected. The NDIR outputs a detection signal (analog signal) which generates a peak whose area is calculated by a data processor.

3.2.3.2 Measurement of IC (Inorganic Carbon)

A sample of 2000 µl is introduced with an injector into an IC reactor vessel where carrier gas is flowing in form of tiny bubbles in the solution acidified by IC reagent. Only IC component in the sample is decomposed to become CO₂, which is detected by the NDIR. The IC concentration can be determined in the same procedure as the TC concentration. Carbon in the form of carbonate and hydrogen carbonates can be measured as IC.

3.2.3.3 Measurement of TOC (Total Organic Carbon)

TOC concentration can be obtained by subtracting the IC concentration calculated in section 3.2.3.2 from the TC concentration calculated in section 3.2.3.1.

3.3 Experimental Procedures

Ultrafiltration experiments were carried out in a 400 ml stirred cell (Figure 3.1). A 300 ml solution of polyelectrolyte, chromate ion, sulfate ion or nitrate ion was placed in the stirred cell which was submerged into the acrylic plexi-glass plastic box having circulating water to control the solution temperature. The experiments were run at 30 °C. The solution was stirred at a speed of 250 rpm with a pressure drop of 60 psi across the membrane using nitrogen gas. Approximately 200 ml of the solution was filtered through the membrane as permeate to produce eight samples of 25 ml each. pH of initial solution was measured about 8.5. Fluxes were determined during the run by timing and weighting samples of permeate. The permeate samples were analyzed for concentrations of polyelectrolyte, chromate, sulfate or nitrate. The rejection of chromate, sulphate, or nitrate was determined by analyzing the sample at the midpoint where 100 ml of permeate have passed through the membrane. By knowing permeate concentration during run, the retentate (the solution not passing through membrane) concentrations at any point in the run were calculated from a material balance. In this work, ratios of QUAT to the anions were varied by fixing the QUAT concentration in retentate approximately 200, 300 and 400 mM to determine rejection of anions.

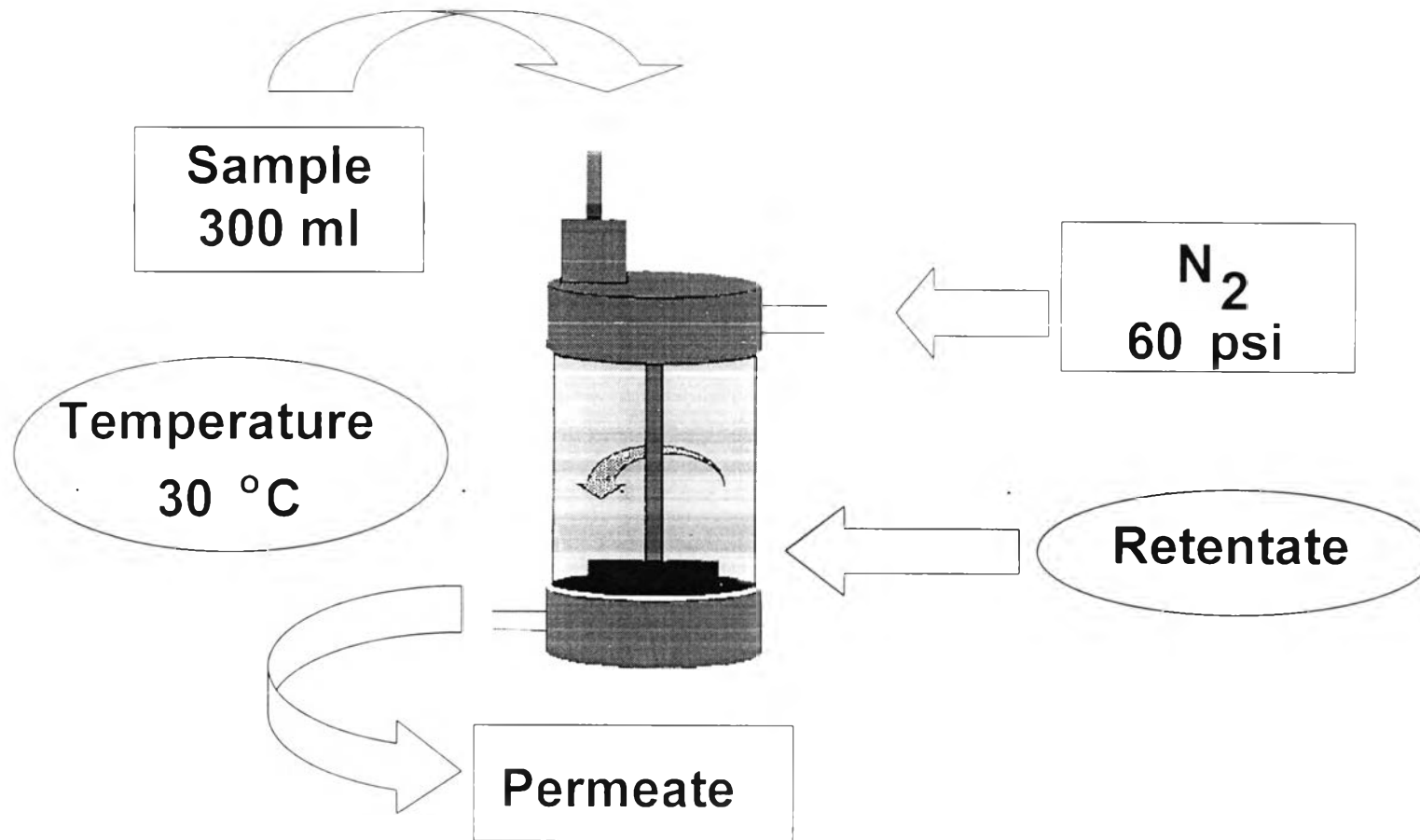


Figure 3.1 Diagram of ultrafiltration apparatus.