CHAPTER III

METHODOLOGY

3.1 Materials

3.1.1 Ion Exchange Resin

The ion pair selected was Na^{+}-H^{+} because of its simplicity and convenience of analysis for hydrogen ions. Dowex50-X8 was selected as the ion exchange resin because it behaves like a strong acid and the resin beds are spherical in shape.

The ion exchange resin used in all experiments was a H type of strong acid synthetic resin, manufactured by Dow Chemical Co., Ltd. under the trade name Dowex 50-X8. The ion exchange resin was needed to be pretreated before it was used in the experiments. It was washed several times using deionized water to remove chemical impurities. Then the resin was dried in an oven for over 2 hours at 70 C and was put in a dessicator.

3.1.2 Other Chemicals

The aqueous solution for the experiments was prepared by adding appropriate amounts of sodium chloride (AR grade) into deionized water to have sodium ion concentrations of 0.1, 0.2 and 0.4 N, respectively. The regenerating solution for the experiment was prepared by adding 37% hydrochloric acid in deionized water to have a hydrogen ion concentration of

0.4 N. Both NaCl and HCl used in these experiments were A.R. grade which were obtained from the Merck Corporation.

3.2 Experimental Setup

Figure 3.2 illustrates the experimental setup of the ion exchange column used in this study. The ion exchange operations were carried out in a standard experiment setup. Three storage tanks containing deionized water, hydrochloric acid solution and sodium chloride solution were used. A positive displacement metering pump (Masterflex) was used to provide an accurate and stable superficial velocity in the column during the regeneration, backwash and adsorption steps. A glass column having a 28.5 mm ID and 250 mm height was packed with the resin. The resin was retained in the column by placing the 60 mesh stainless screen on the top portion of the column and the low portion was equipped with sintered glass. The void fraction in the compacted bed is 0.41. Three shut-off valves and one four-way selection valve were used so that any of the three liquids could be pumped through the bed, either in upflow or downflow. Two pH electrodes were mounted at the bed inlet and exist for measuring the pH value as a function of time. A computer is used for data acquisition. The height of the resin bed was monitored by means of the scale mounted outside of the column.

3.3 Experimental Procedure

3.3.1 Batch Experiment

The ion exchange experiments were conducted in a beaker of 1000 cm^3 equipped with two acrylic sheets baffles for obtaining complete mixing. The

content in the beaker was kept well mixed with a magnetic stirrer. For each experiment, 600 cm^3 of prepared aqueous solution was added to beaker. The 70 g of pretreated resin was put into the beaker. The pH values were measured periodically as a function of time. All experiments were performed at ambient temperature.

A schematic diagram for the equipment used in this experiment is shown in Fig. 3.1.

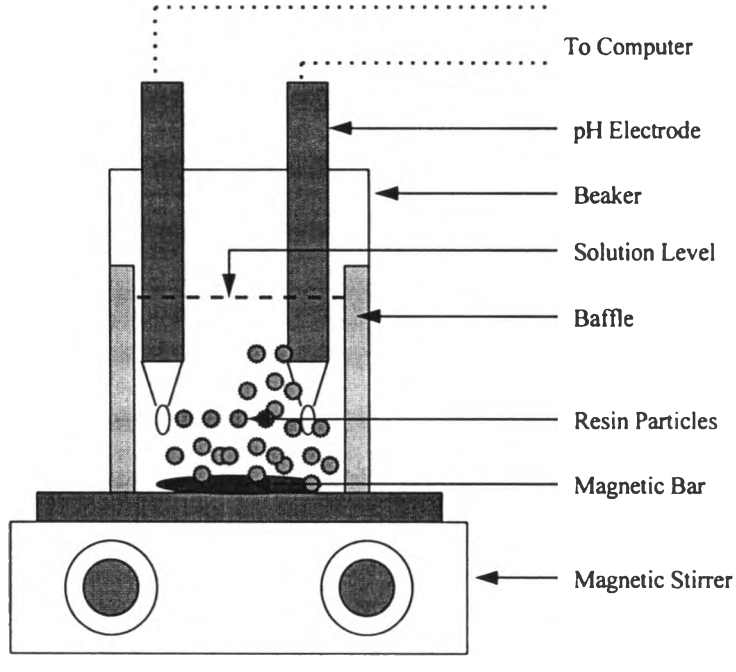

Figure 3.1 Schematic diagram of experimental apparatus for batch operation.

3.3.2 Dynamic Continuous Flow Experiment

A schematic diagram for the equipment used in the continuous flow is shown in Figure 3.2.

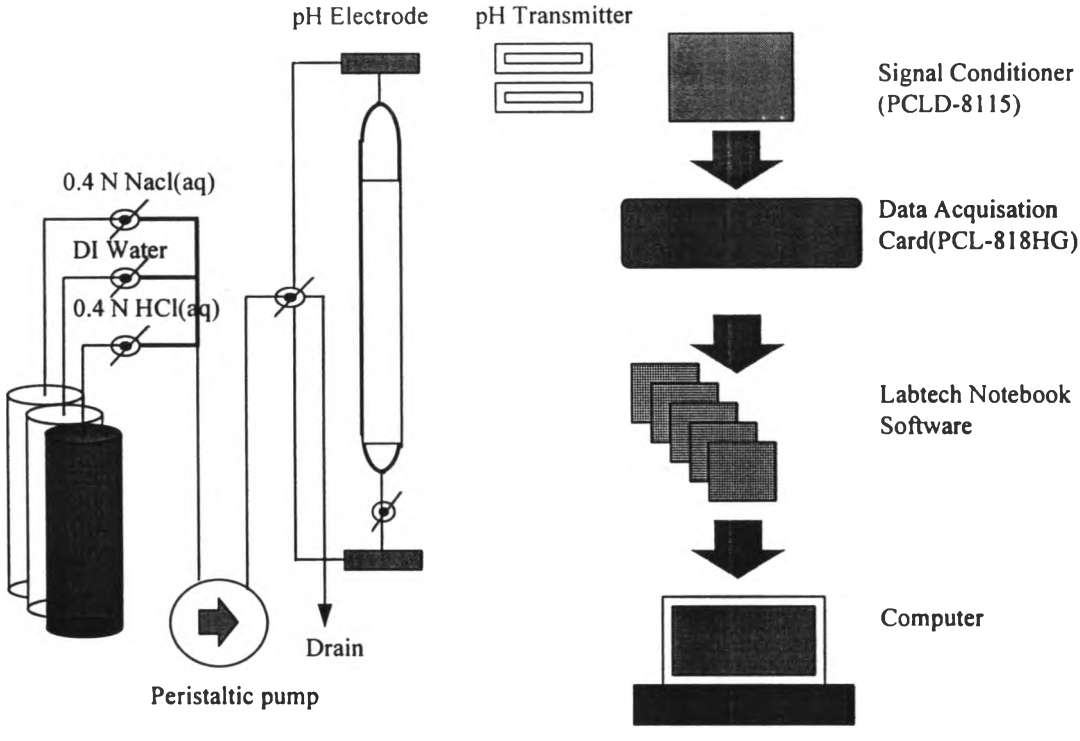

Figure 3.2 Schematic diagram of experimental apparatus for continuous flow operation.

The unit was designed to operate in either upflow or downflow by rotating the four-way selector valve. Only one of the fluid selector valves is open to select either water, the HCl solution, or the NaCl solution as needed. The two valves at the inlet and outlet and the bed should be in the open position.

Before performing any experimental runs, the desired flow rate was calibrated by pumping deionized water through the resin column.

No Adsorption Experiment with Upflow Direction

1.. Switch the three shut off valve to 0.4 N HCl to allow the resin bed to be contacted with 0.4 N HCl in upflow. Continue to monitor the pH of the effluent unit no further change is observed so the bed becomes entirely in its active form.

2. Switch the three shut-off valve to deionized water to flush the residual hydrogen ions from the bed in upflow. The process is ceased when the residual hydrogen ions are completely purged from the bed with no change in the pH of the effluent.

3. Again, switch the three shut-off valve to 0.4 N HCl to allow the resin bed to be contacted with 0.4 N HCl in upflow direction for which 0.4 N HCl act as a tracer. The process is stopped when the pH values of influent and effluent are equal.

Adsorption Experiment with Upflow Direction

4. Repeat step 2 as a backwash step.

5.Switch 3 shut off valve to the sodium chloride solution and then use Labtech Notebook software for data acquisition and continue until the pH of the effluent shows no further change in which all sites are saturated with sodium ions.

No Adsorption Experiment with Downflow Direction

6.Repeat steps 4, 5 and 6 but in downflow direction.

Adsorption Experiment with Downflow Direction

7. Repeat steps 7 and 8 but in downflow direction.