

CHAPTER III

EXPERIMENTAL SETUP

3.1 Equipment Setup

The experiment setup for the continuous operation is shown in Figure 3.1. The solution feeding selection was composed of three storage tanks for deionized water, HCl and test solution. The test solution used here was NaCl, MgCl₂, or CaCl₂, or a mixture of CaCl₂ and MgCl₂. Each storage tank was equipped with a three-way shut-off valve. A positive displacement-metering pump obtained from the Masterflex Co., Ltd. was used to adjust the liquid flow rate to the column containing the ion-exchange resin. During the experiment, a flow stream from each tank passed through a four-way flow selection valve. This valve was used to let the flow pass through the bed, either in the upflow or downflow direction. Two pH electrodes obtained from Cole-Parmer Co., Ltd. (model H-05993-70 and H-05656-10) were mounted at the inlet and exit of the column to measure pH as a function of time. In this work, a glass column having a 28.5 mm ID and 250 mm high was packed with Dowex50-x8 resins. The resins were retained in the column with a 60 mesh stainless steel screen on the top and bottom parts of the column. The void fraction in the compacted bed before the fluidization was 0.41. The pH value obtained from the electrode was sent to a DA&C hardware, which converts the analog signals into the digital form so that a computer can process them. The computer then recorded and performed a calculation on the acquired data by using the Labtech Notebook software, which was obtained from Labtech Co., Ltd. The DA&C hardware consisted of a signal conditioning circuit (a writing terminal board; PCLD-8115) and a data acquisition card (PCL-818HG).

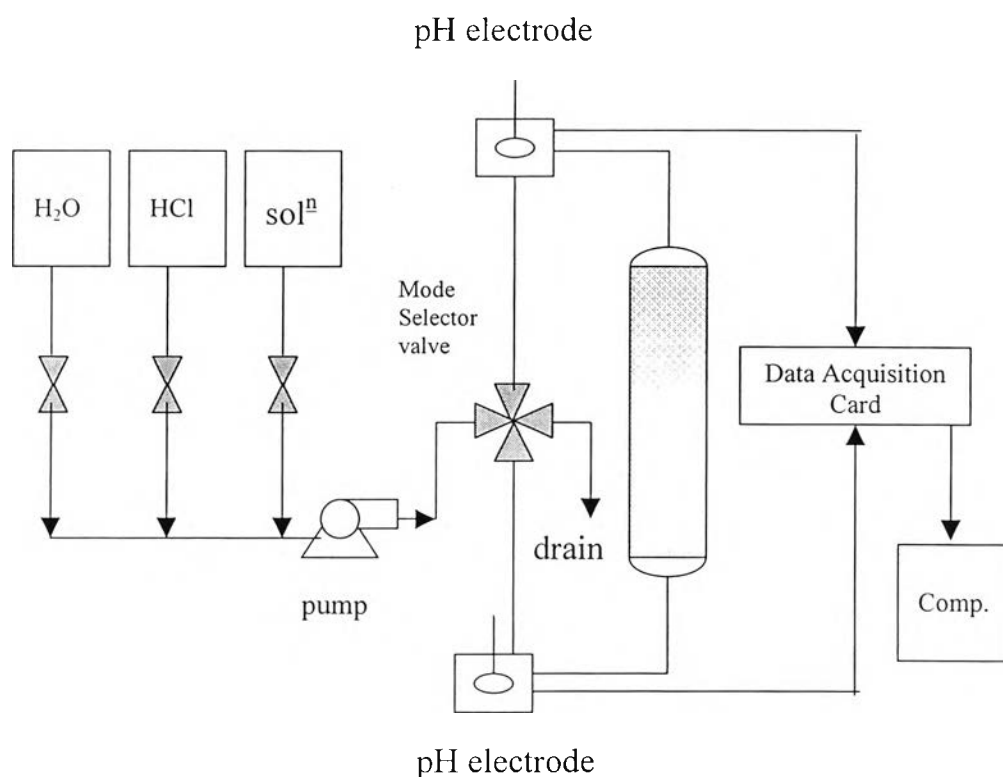


Figure 3.1 Schematic of the continuous operation

3.2 Experimental Procedure

3.2.1 Resin Preparation

The ion-exchange resin employed here was the H^+ type Dowex50-X8 obtained from Dow Chemical Co., Ltd. The resin is a strong acid cationic ion-exchange resin. The ion-exchange resin was pretreated before it was used in the experiments. The pretreatment started with equilibrating the resin with 600 ml. of 0.4 N HCl . Subsequently, the resin was washed several times with deionized water to remove chemical impurities and excess H^+ . Then, the hydrated resin in the H^+ form was dried in an oven at 70 $^{\circ}C$ for 7 hr.

3.2.2 Ion Exchange Experiments

3.2.2.1 pH Calibration

Before starting the experiment, each pH electrode was calibrated against buffer solutions with pH values of 1, 4 and 7. The buffer solutions were obtained from Merck Co., Ltd.

3.2.2.2 Batch Experiment

The experimental setup for the batch experiment operated at room temperature is shown in Figure 3.2. 120 g of the hydrated resins were placed into a beaker equipped with four baffles. The beaker was then kept well mixed with a magnetic stirrer. 600 ml of 0.2 N testing solution, which could be NaCl, MgCl₂, CaCl₂ or a mixture of CaCl₂ and MgCl₂ solution, was then added. Then, pH value was measured as a function of time until the pH was constant, which means the system is in equilibrium. For the mixture of CaCl₂ and MgCl₂ solution, the sample was taken every 30 seconds to determine Ca²⁺ and Mg²⁺ concentration by an Atomic Absorption Spectroscopy (AAS).

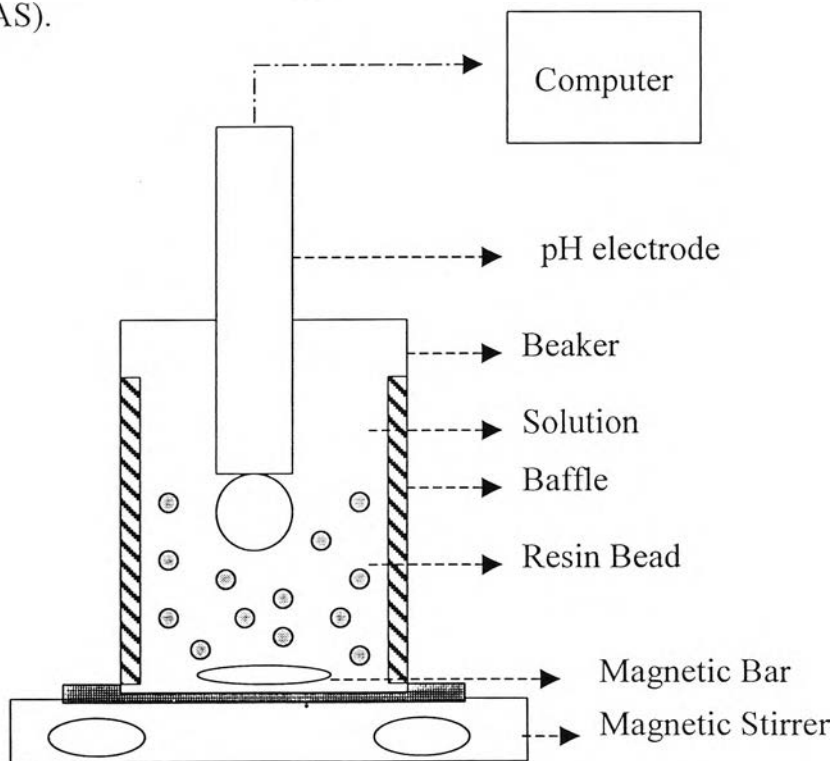


Figure 3.2 Experimental setup for batch operation

3.2.2.3 Continuous Flow Experiment

All the continuous flow was carried out in the laboratory-scale glass column at room temperature. The operation of the continuous flow using here was the fluidized-bed operation, and two sets of the experiment were performed here. The first one was continuous flow with *no* adsorption on the resin. The other was the same with the adsorption operation.

No adsorption experiment

0.4 N HCl solution was fed into the column packed with Dowex50-8X in the upflow direction. The pH value was measured until the pH of the influent and effluent streams were the same. Then, deionized water was fed into the system to remove the excess ions from the bed in the downflow direction. Again, pH of the feed and the effluent were used as an indicator for the complete removal of H^+ . The no-adsorption operation was then started by feeding 0.4 N HCl solution into the bed in the upflow direction, and the pH value of the feed and the effluent were used to check whether the system was in steady state.

Adsorption experiment

First, 0.4 N HCl solution was fed into the column in the upflow direction until the resin was saturated with the acid. Then, deionized water was fed into the system to remove the excess hydrogen ions from the bed in the downflow direction until the pH of the influent and effluent were the same. Finally, 0.2 N of testing solution was fed into the system in the upflow direction. The pH value was measured until the pH values of both influent and effluent streams were the same, which means the adsorption is in equilibrium.

For the mixture solution, the sample was taken from the effluent every 30 min. The samples were analyzed for the concentration of Ca^{2+} and Mg^{2+} ions by the AAS (atomic absorption spectroscopy).