

## CHAPTER III

### EXPERIMENTAL METHODS

#### 3.1 Materials

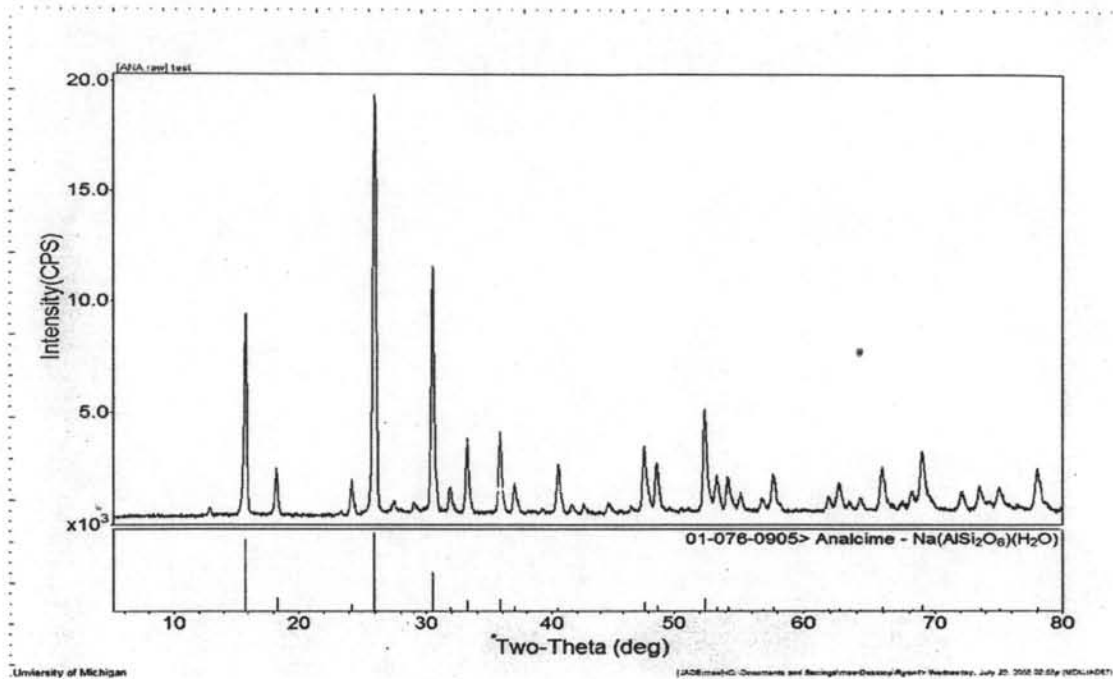
Naturally occurring analcime was obtained from Mt. St. Hillaire, (Quebec through Ward Science). The analcime is a hydrated sodium aluminosilicate with a density of 2.22-2.29 g/cm<sup>3</sup> at 20 °C. It appears to be colorless to white crystals. The crystals were crushed using mortar and pestle, then sieved successively through sieve sizes 150 µm, 75 µm, 53 µm and 38 µm until all particles were < 38 µm.

Hydrochloric acid supplied by Fisher Scientific (37% wt) was used as the acidizing fluid in this study. Deionized water was used as the diluting agent for the preparation of each acid solution. Hydrofluoric acid obtained from Aldrich was used to verify the analcime purity and amorphous silicate gel precipitates. Silicon, aluminum and sodium reference standard solutions for atomic absorption spectrophotometry (AAS) were purchased from Fisher Scientific.

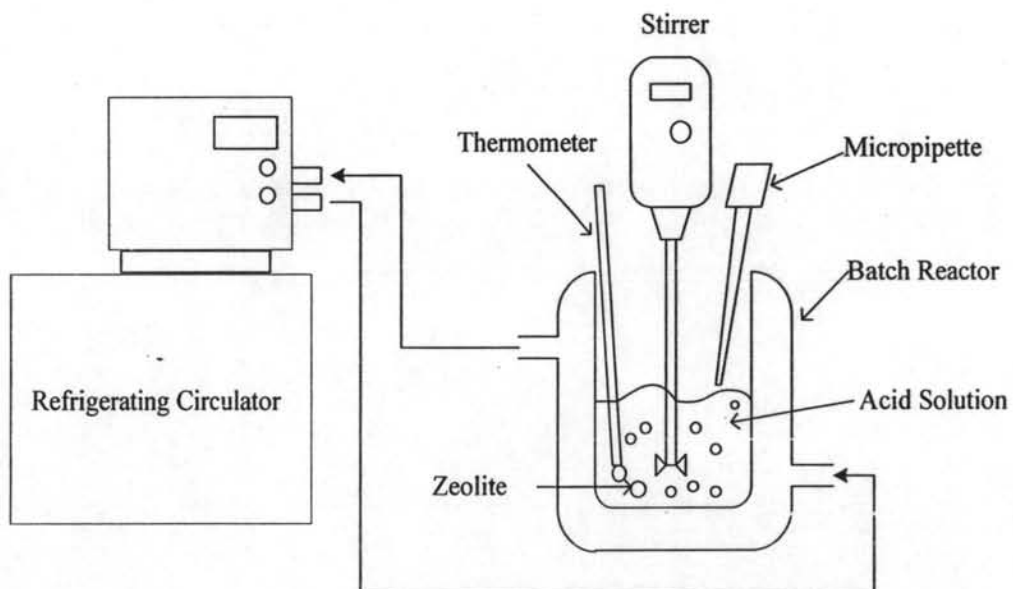
Citric acid supplied by Sigma-Aldrich was used as an alternative acidizing fluid and chelating agent in this study. Citric solution was prepared by dissolving in deionized water. Potassium hydroxide and hydrochloric acid were used to adjust the pH to the desired value.

#### 3.2 Analcime Characterization

Three different samples were prepared by dissolving approximately 0.1 g of analcime in 50 mL of a 2.5 M HCl/0.5 M HF mixture in an oven at 70 °C overnight. Each sample was drawn through a 0.2 µm membrane filter, diluted with deionized water, and analyzed using Perkin Elmer 3100 Atomic Absorption Spectrophotometer (AAS). The molar ratio of Si:Al:Na in analcime was calculated to be 2.25:1.00:0.34. The compositions of Si, Al, Na, and O in each sample were analyzed by using a Philips XL30 FEG scanning electron microscope equipped with EDXS and ICP/MS. The crystal structure of analcime was analyzed by XRD.



**Figure 3.1** Crystalline structure of analcime analyzed by XRD.

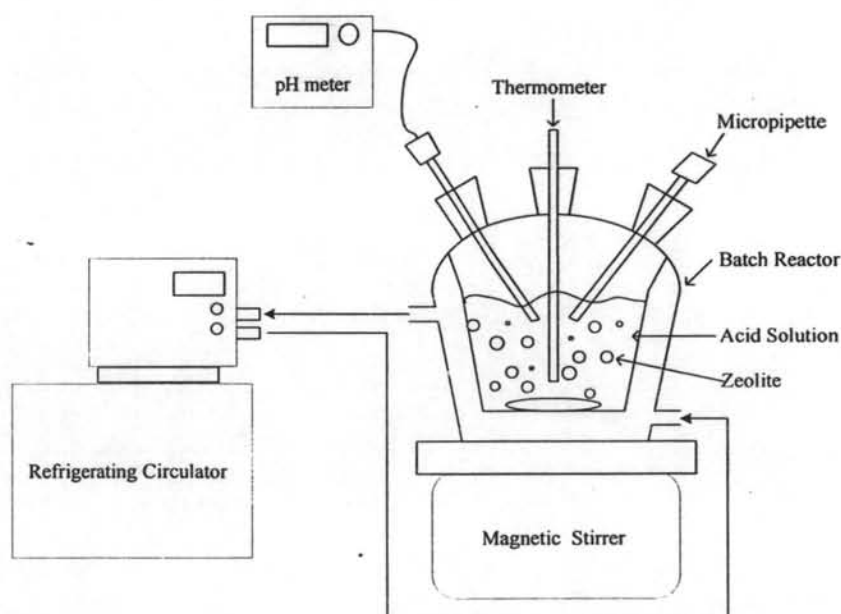


**Figure 3.2** Experimental batch apparatus for analcime dissolution experiments.

### 3.3 Dissolution and Precipitation Experiments

Analcime dissolution reactions were carried out in different concentrations of hydrochloric acid using a batch reactor shown schematically in Figure 3.2. Different amounts of analcime were dissolved in 150 mL of acid solution at 5, and 25 °C. Sample (~2 to 4 mL) were taken using micropipettes and drawn through polypropylene filters (pore size = 0.2  $\mu\text{m}$ ) at short time intervals. Filtration of the suspended particles would stop the dissolution reaction. Hence, the filtrate samples were analyzed for dissolved Si, Al, and Na using AAS in order to monitor the reaction progress. The stirring rate used in each experiment was approximately 500 rpm. Initial concentrations of HCl acid ranging from 0.1 to 8 M were prepared and reacted with analcime each separately. Dissolution in citric acid were studied as the same method.

Silicate precipitation was studied in hydrochloric and citric acid using a closed bath reactor shown in Figure 3.3. Different amounts of analcime were dissolved in 300 mL of acid solution at 5, and 25 °C. The method for taking and analyzing the sample are the same as the method for dissolution experiments.



**Figure 3.3** Experimental batch apparatus for precipitation experiments.