

CHAPTER III EXPERIMENTAL

3.1 Materials

Naturally occurring analcime was obtained from Mt. St. Hillaire, (Quebec) through Ward Science. The density of the particles was about 2.22-2.29 g/cm³ at 20 °C. The crystals were crushed and sieved to <38µm size.

The acids used for the dissolution and precipitation studies were, Hydrochloric acid (37%), hydrobromic acid (46.8%), hydriodic acid (65%), nitric acid (70%) and sulfuric acid (95%). All were supplied by Fisher Scientific or Sigma Aldrich. Deionized water was used as the diluting fluid for preparing the acid solutions. Silicon and Aluminum were characterized by a Perkin Elmer 3100 AA Spectrophotometer.

3.2 Equipments

3.2.1 Jacketed vessel with constant temperature bath and circulator (Figure 3.1)

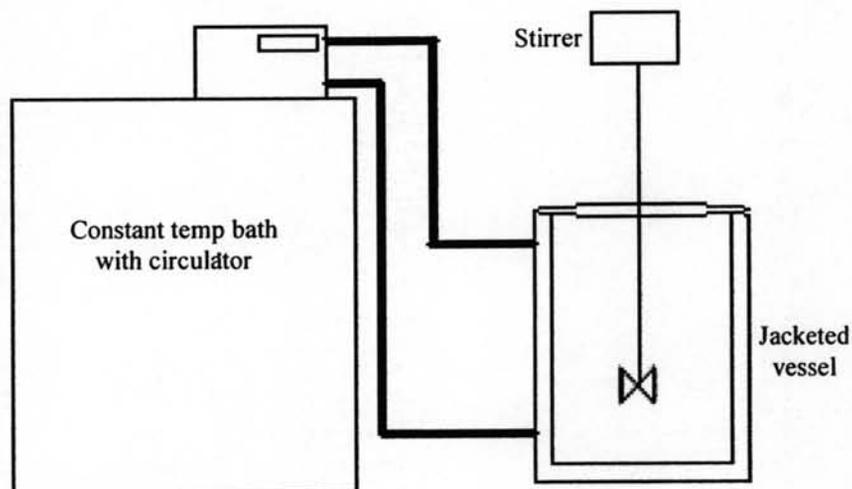


Fig 3.1 Experimental setup

3.2.2 Perkin Elmer 3100 AA spectrophotometer

3.2.3 Philips XL 30 FEG SEM with EDAX. This was used for SEM images and to characterize the surface of the undissolved particles for their composition.

3.2.4 A filter holder assembly was used to filter the particles and characterize through SEM. The assembly included polypropylene filter placed in a filter holder. The filter holder assembly was attached to a syringe.

3.3 Methodology

3.3.1 Initial Dissolution Rate Experiments

Analcime dissolution experiments were carried out in different concentrations of acids in a jacketed batch reactor. For the initial dissolution rate experiments, solutions were prepared at different $[H^+]$ concentrations and the dissolution reactions were carried out at 5 °C at a stirrer speed of 500 rpm. Samples were taken out of the reaction mixture at very short time intervals (10-15 sec) and till a very short reaction time (1-2 min), using a hand pipette and were immediately filtered using .20 μm filters. The filtrates were diluted to about 50 times with 3000 ppm KCl solution and then analyzed using AAS to get the concentration time trajectories of Si and Al at different acid molarities. The acids used for the dissolution rate study were, hydrochloric, hydrobromic, and nitric acids.

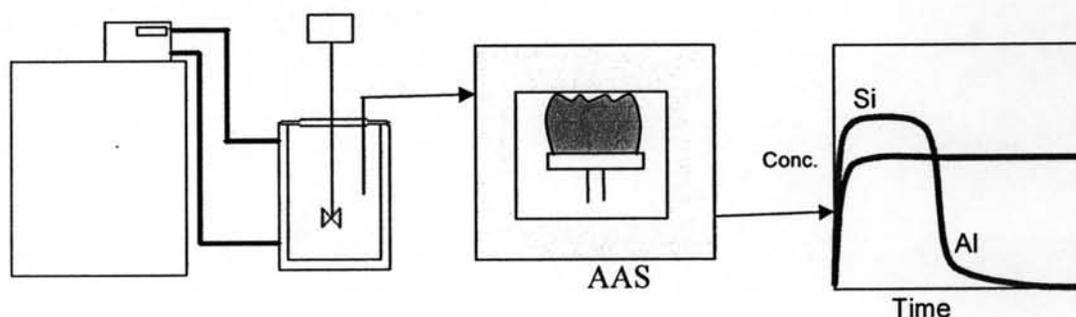


Fig 3.2 Experimental procedure

3.3.2 Silica Precipitation Experiments Under Base Case Conditions

All the precipitation experiments were carried out at a $[H^+]$ concentration of 8M. Samples were drawn out of the vessel using pipettes at predetermined time intervals. A 2 μ m filter syringe assembly was used to filter out the undissolved particles. The sample was then diluted with KCl and analyzed using AAS. The acids used for the precipitation experiments were hydrochloric, hydrobromic, hydriodic, nitric and sulfuric acids.

3.3.2.1 Sample preparation for SEM-EDAX

The partially dissolved particles were filtered out from the reaction mixture at the three stages of the precipitation experiments, i.e., dissolution, plateau and during the precipitation. After being filtered, the particles were washed with water to remove any traces of acid, dried and analyzed using SEM-EDAX.

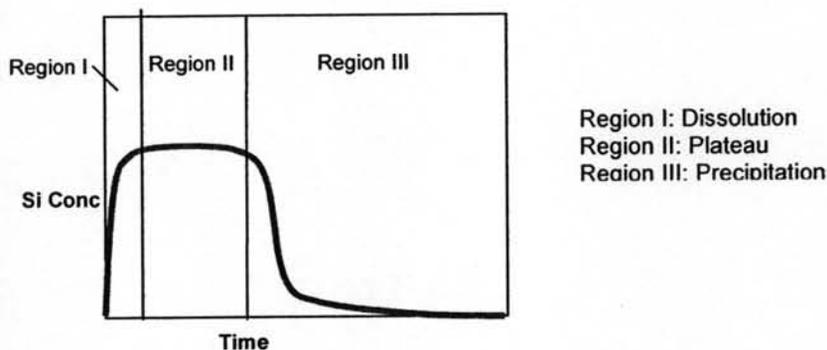


Fig 3.3 Trajectory of Silica concentration during base case conditions