CHAPTER III
EXPERIMENTAL

3.1 Materials

The silica used in this study was obtained in the form of Sodium Metasilicate Nonahydrate (SMN); 44-47.5% total solids, $Na_2O_3Si \cdot 9H_2O$, M.W. 284.19, by Thermo Sci Acro Organics. The Si content in SMN was determined using Inductively-Coupled Plasma/Mass Spectrometer (ICP/MS) and was found to be 0.104 ± 0.001 gSi/gSMN.

Analytically pure 35-37% wt. trace metal grade HCl solution was provided by Fisher Chemical. Laboratory grade salts were also supplied by different dealers as listed in Table 3.1. Ultrahigh purity, deionized water provided by a MilliQ system was used as a diluent in the preparation of all solutions.

Table 3.1 Salts used for this research.

Chemical	Chemical formular	Molecular weight	Supplier
Sodium Chloride	NaCl	58.44	Fisher Chemical
Cesium Chloride	CsCl	168.36	Sigma-Aldrich
Magnesium Chloride	$MgCl_2 \cdot 6H_2O$	203.30	Fisher Chemical
Calcium Chloride	$CaCl_2 \cdot 2H_2O$	147.02	Fisher Chemical
Aluminium Chloride	$AlCl_3 \cdot 6H_2O$	241.42	Fisher Chemical
Sodium Nitrate	$NaNO_3$	84.99	Aldrich Chemical
Sodium Bromide	NaBr	102.9	Sigma Chemical
Sodium Iodide	NaI	149.89	Fisher Chemical

3.2 Experimental Procedures

First, 13.82 grams of SMN were dissolved in a cooling jacketed 3-pronged glass reaction containing a 70mL of deionized water for approximately 50 minutes. The solution was magnetically stirred at a stirring rate of 500rpm and cooled down by the circulation system with a constant coolant temperature of 5.0°C. Once completely dissolved, a hydrochloric acid solution and a solution of make-up DI water and salt cooled down to 5.0°C were added into the reactor to initiate the reaction. The final properties of this solution were 170mM $Si(OH)_4$, 1M HCl, 1M salt, and 300mL total of solution.

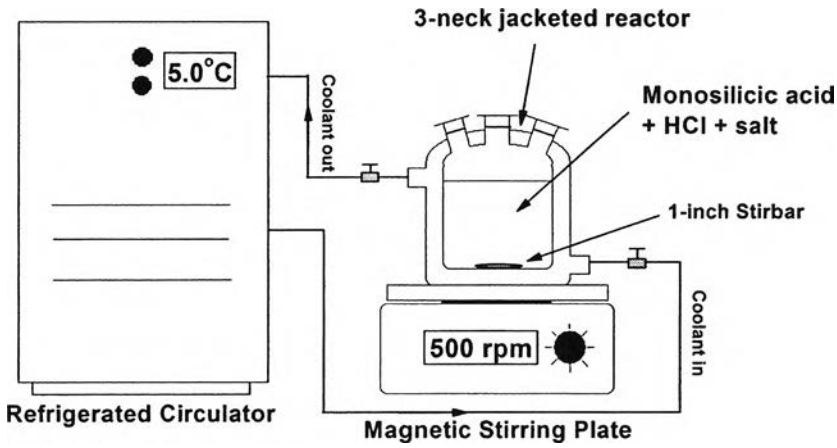

Figure 3.1 Experimental setup.

3.3 Characterization techniques

3.3.1 Si Content in Solution Using Inductively-Coupled Plasma/Mass Spectroscopy (ICP/MS)

Samples were manually obtained using micropipettes and drawn through polypropylene-membrane filters (d_{pore}=0.2μm) at short interval times and placed into 50mL sample tubes. Filtrate solutions were diluted twice by DI water with a dilution ratio of 1 to 25 to reach the appropriate concentration for compositional analysis using ICP/MS (ELAN9000, Perkin Elmer).

3.3.2 Silica Particle Growth Using Dynamic Light Scattering (DLS)

At particular times, samples were taken out of the reactor, put into a vial, and analyzed for silica particle size measurement using DLS (Nano ZS, Malvern) immediately. The DLS measurements were also carried out at a constant temperature of 5.0°C. Intensity-mode particle diameter DLS measurements were used to calculate the mean particle size. The particle size results were used for modeling the particle growth profile.

3.3.3 Monosilicic Acid Remaining in Solution Phase Using Molybdate-Blue Method and Ultraviolet-Visible Light Spectroscopy (UV-Vis)

Samples were withdrawn at short reaction times and transferred to 50mL sample tubes. The solutions were diluted by DI water using dilution ratios of 1 to 25 and 2 to 45, respectively. Molybdate solution was then added, causing the color of solutions to change from clear to blue. A UV-Vis spectrometer (Cary100, Varian) was used to determine the concentration of monosilicic acid remaining, which was quantified in terms of "molybdate reactive silica" according to ASTM D859-05.