



CHAPTER III EXPERIMENTAL

3.1 Materials

- Stearic acid >98.5% purity was purchased from Sigma
- Calcium hydroxide 99.995% purity was purchased from Aldrich
- Deionized water
- Absolute ethanol >99.8% purity was purchased from Italmar
- Acetone >99.9% purity
- Hydrochloric acid >37% purity
- Sodium hydroxide 98% purity
- Dimethyldodecylamine oxide 99% purity was purchased from Aldrich
- Disodium ethylenediaminetetraacetate was purchased from CARLO ERBA
- Sodium Chloride 99% purity was purchased from RCI Labscan

3.2 Equipments

- GAST Manufacturing Inc., Model DOA_P504_BN, Vacuum pump
- Fisher Scientific, Model 285A, Vacuum oven
- METTLER TOLEDO, pH meter
- METTLER TOLEDO, Balance
- MERMMERT, Water Bath (70°C and 25°C)
- Varian, Model SpectraAA 300, Atomic Absorption Spectrometer (AAS)
- Cole Parmer, Syringe pump
- Nylon membrane filters 13 mm, 0.2 μm was purchased from Vertical® Thailand

3.3 Methodology

3.3.1 Soap Scum Preparation

Soap scum or calcium stearate can be synthesized from the reaction between calcium hydroxide and stearic acid in the ratio of 1:1. Calcium hydroxide was dissolved in deionized water while stearic acid was dissolved in ethanol. Then stearic acid solution was mixed with calcium hydroxide solution. Then the soap scum will form as a white solid. After that leaves it for 1 day to reach equilibrium. Then the precipitate was filtered by using a 0.2 micron nylon membrane and follow by rinsed with water, ethanol and acetone in order to remove the excess calcium ions and unreacted stearic acid. Finally, the precipitate was dried in a vacuum oven at 30°C for 3 hours.

3.3.2 Solution Preparation

For both equilibrium solubility and dissolution rate of calcium soap scum was studied by using amphoteric surfactant (DDAO). Therefore, 0.1 M DDAO was mixed with various NaCl concentrations (0.0001 M – 0.1 M) or mixed with both 0.1M Na₂EDTA and 0.0001 M NaCl. Then, all of the samples were equilibrated for 1 night.

3.3.3 Soap Scum Dissolution

The samples were adjusted to pH 4-11 by using NaOH and HCl. Then an excess amount of synthesized calcium soap scum was added to each sample at the same amount. After that the solution was heated to around 70°C in a water bath for 3 hours or until all added soap scum dissolved. Next all samples were equilibrated at 25°C in the temperature-controlled water bath and let all samples equilibrated at least 1 week with routine shaking. Then the solution was filtered by using a 0.2-micron nylon filter membrane. Finally, the clear solutions were analyzed by atomic absorption spectrometer (AAS) in order to find the concentration of dissolved calcium ion in sample solution.

3.3.4 Dissolution Rate of Soap Scum

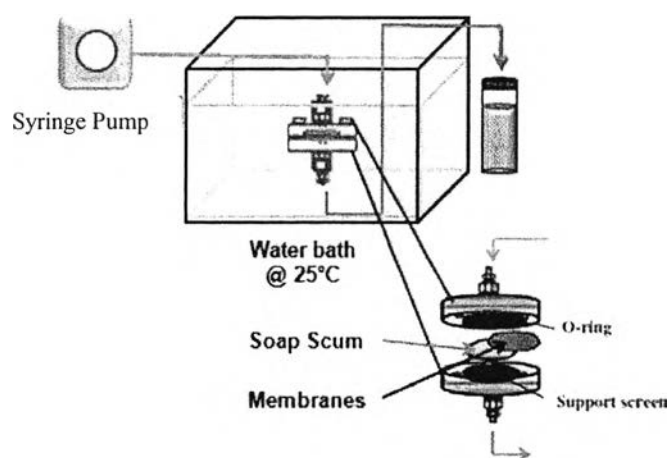


Figure 3.1 Flow cell apparatus

The dissolution rate of calcium soap scum was studied by using a flow cell apparatus (Figure 3.1) which made from a 25-mm Millipore Teflon filter holder. A 0.05 grams of calcium soap scum was placed between 0.22-micron nylon membranes. Then the flow cell was immersed in the temperature-controlled water bath (25°C). Samples were adjusted to pH 4, 7 and 11 by using NaOH and HCl and then the sample was injected through the flow cell apparatus with a syringe pump at a constant flow rate (60 ml/hr). Finally, the sample was collected every 5 minutes until 30 minutes and the concentration of calcium ions in samples were analyzed by using AAS.