CHAPTER III
EXPERIMENTAL

3.1 Materials

- Absolute ethanol >99.8% purity was purchased from Italmar
- Acetone (A.C.S grade) was obtained from J.T. Baker
- Calcium chloride 99% purity was purchased from J.T. Baker Chemicals B.V.
- Deionized water
- Dimethyldodecylamine oxide (DDAO) >99% purity was purchased from Sigma-Aldrich
- Disodium ethylene diamine tetraacetate (Na_2EDTA) 99% purity was purchased from Carlo Erba
- Hydrochloric acid 99% purity was obtained from Lab Scan
- Magnesium chloride >99% purity was purchased from Sigma-Aldrich
- Sodium hydroxide 98% purity was obtained from J.T. Baker
- Stearic acid >98.5% purity was purchased from Sigma-Aldrich
- Tetrasodium glutamatediacetate (Na_4GLDA) was obtained from Dissolvine ® GL-38

3.2 Equipment

- Cole Parmer, Model 74900-00, Single-syringe infusion pump, 115 VAC
- Fisher Scientific, Model 285A, Vacuum oven
- GAST Manufacturing Inc., Model DOA_P504_BN, Vacuum pump
- MERMMERT, Water Bath (70°C and 25°C)
- METTLER TOLEDO, pH meter
- METTLER TOLEDO, Balance
- Nylon membrane filters 13 mm, 0.2 μm was purchased from Vertical® Thailand

- Varian, Model SpectraAA 300, Atomic Absorption Spectrophotometer (AAS)
- Whatman, Model 1980-002, Stainless Steel Syringe Filter Type Membrane Filter Holder, 25mm diameter

3.3 Methodology

3.3.1 Mixed Soap Scum Preparation

Mixed calcium and magnesium stearate at molar ratios 1:1 and 4:1 are models in this experiment which can be synthesized from the reaction between mixed calcium and magnesium chloride and stearic acid following stoichiometric ratio. The stearic acid dissolved in ethanol and then mixed with a clear solution of calcium chloride and magnesium chloride following the ratios. The solutions were left over night to complete the reaction. Then, the soap scums were generated as white precipitate. After that, the precipitate was filtered by using a 0.2 micron nylon membrane and rinsed with water, ethanol and acetone in order to remove the unreacted stearic acid and excess calcium and magnesium ions. Finally, the soap scum was dried in a vacuum oven at 30°c for 3 hour.

3.3.2 Mixed Soap Scum Equilibrium Solubility Experiments

The equilibrium solubility of mixed calcium and magnesium soap scums was analyzed by using amphoteric surfactant (DDAO) with different chelating agents (Na_2EDTA and Na_4GLDA) at 25°C. The HCl and NaOH solutions were used to adjust pH value of 4-12 in solutions. An excess amount of each synthesized mixed soap scum was added in mixed solution containing DDAO and various chelating agents at the pH of interest. Then, the mixed solutions were heat up to 70°C in a water bath for 3 h. After that, they were equilibrated in temperature-controlled water bath at 25°C with daily shaking at least 1 week. Then, the mixtures were filtered using a 0.2 micron nylon filter membrane to separate the remaining undissolved soap scum. Finally, the clear solutions were taken for analyzing calcium and magnesium concentration by the atomic absorption spectrophotometer (AAS) (SpectrAA-300,

Varian). The average data were obtained from at least 3 times with less than 1% error.

3.3.3 Soap Scum Dissolution Rate Experiments

The rate of mixed soap scum experiment was done by differential reactor (a flow cell) which was made from 25-mm Millipore Teflon filter holder (Itsadanont et al, 2013). The flow cell was connected by syringe pump (Cole-Parmer®, 74900-00) at a constant flow rate of 1 mL/min to inject the solution containing 0.1 M DDAO and 0.1 M chelating agent at pH of 11. The amount of 0.095 g of the mixed soap scum sample was placed in between two 0.22-micron nylon membranes in a filter cartridge (Whatman, 1980-002). The flow cell was merged in a temperature-controlled water bath, which was set at a constant temperature of 25°C. The sample were collected every 5 min until 30 min and determined calcium and magnesium concentration by AAS.

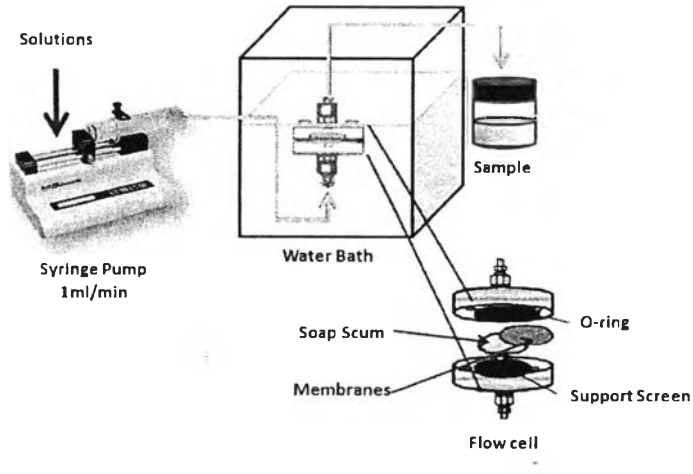

Figure 3.1 Flow cell apparatus.

3.4 Analysis Section

3.4.1 Atomic Absorption Spectrophotometer

Firstly, AAS, Varian model, was used to confirm the ratios of calcium to magnesium concentrations after synthesis and to determine the quantity of calcium and magnesium stearate which were dissolved in various solutions. The 1000 ppm

standards of calcium and magnesium were prepared for providing calibration curve. The samples were pipetted and digested by 50% aqueous nitric acid solution. Then, the solutions were heated in water bath at 70°C. After that, they were left until cool and diluted by distillation water into the range of calcium and magnesium standard.

3.4.2 Fourier Transform Infrared Spectroscopy (FT-IR)

FT-IR transmission mode was used to analyze the functional group of mix soap scum samples. The method for preparation of all samples involves mixing each sample (about 5% by weight) with an IR transparent material (typically KBr). The mixing can also be done with a mortar and pestle but not as well and pressing a pellet. A pellet involves pressing the prepared mixture with a hydraulic or hand press. Then, the sample was placed into machine. The obtain peak was matched with the reference peak wavenumber.

3.4.3 Particle Size Analysis (PSA)

The particle size was determined the size range, the average and mean size of the particles in a powder or liquid sample. The mixed soap scum samples were investigated the particle size distribution and average diameter.

3.4.4 Scanning Electron Microscope (SEM)

Low vacuum SEM were used to analyze the surface morphology of 1:1 and 4:1 calcium and magnesium stearate. Firstly, the samples were dried in order to trap the humidity out and then the samples were placed on a stub by carbon tape and coated by Platinum. The 1K and 10K magnification were used in each of samples. The focus and brightness were set up and then SEM would be created the picture by catching the single from back scattering electrons with voltage of 15 kV and magnification of 10K.

3.4.5 X-ray Diffractometer (XRD)

XRD was performed by using a Rigaku Dmax X-ray Diffraction, RINT-2200 with Cu tube for generating CuKα equipped with a Ca and Mg filtered CuKα radiation source (λ = 1.542A°) of 40 kV and 30 mV. An exact pattern match

can be found between the unknown and the authentic sample, so chemical identity can be assumed. X-ray diffraction patterns of mixed soap scum samples were obtained. A mixed soap scum sample was pressed on a hollow of glass holder and held in place by glass window. Then, it was scanned in the 2θ range from 10° to 80 ° in the continuous mode with the rate 5°/min. Then, XRD pattern would be used to characterize.