

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 diaminetetraacetate (Na₂EDTA) 99% purity was purchased from Carlo Erba
- Hydrochloric acid was obtained from Lab Scan
- Magnesium chloride 99% purity was purchased from Sigma-Aldrich
- Sodium hydroxide was obtained from J.T. Baker
- Stearic acid 98.5% purity was purchased from Sigma-Aldrich
- Tetrasodiumglutamatediacetate (Na₄GLDA) was obtained from Dissolvine ® GL-38
- Lux, commercial soap was purchased from a supermarket in Bangkok.

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
- METTLER TOLEDO, pH meter
- METTLER TOLEDO, Balance
- MERMMERT, Water Bath (70°C and 25°C)
- Nylon membrane filters 13 mm, 0.2 µm was purchased from Vertical® Thailand

- Varian, Model SpectraAA 300, Atomic Absorption Spectrometer (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

Soap scum synthesized from stearic acid were prepared by dissolved stearic acid in ethanol. But for commercial soap was dissolved in deionized water. After that the dissolved reactant mixed with a clear solution of pure calcium chloride, pure magnesium chloride, 4:1 Ca:Mg solution and natural hard water. 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 reactants. 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

To study the equilibrium solubility of synthesized stearic acid and commercial soap scums, an excess amount of both synthesized soap scum was added in mixed solution containing amphoteric surfactant (DDAO) and chelating agents (Na₂EDTA or Na₄GLDA) at 25°C adjusting the pH around 11. 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) (SpectraAA-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 0.03 g of the each 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. The system schematic was shown in Figure 3.1.

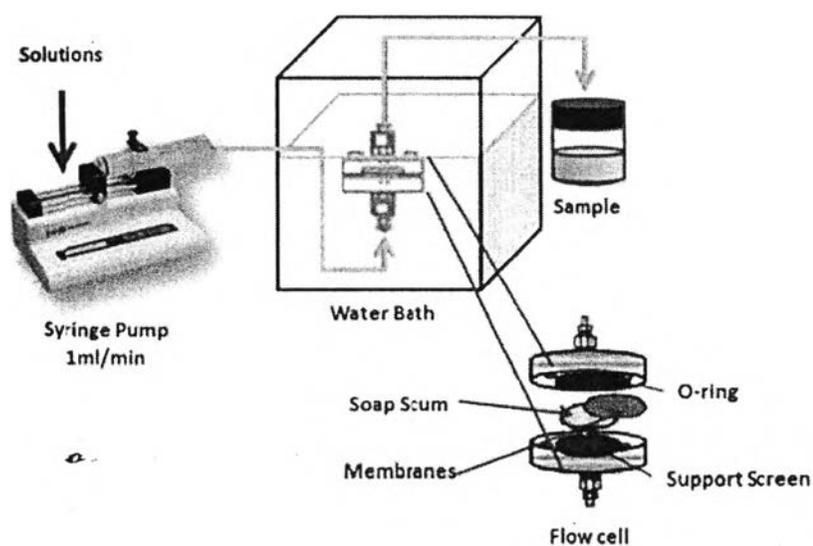


Figure3.1 System schematic of flow cell.

3.4 Analytical Method

The characterization equipments consisted of Atomic Absorption Spectrophotometer, Fourier Transform Infrared Spectroscopy, Particle Size Analyzer, Scanning Electron Microscope and X-ray Diffractometer.

3.4.1 Atomic Absorption Spectrophotometer (AAS)

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)

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 wave number.

3.4.3 Particle Size Analyzer (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 soap scum. 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.

3.3.5 X-ray Diffractometer (XRD)

XRD was performed by using a RigakuDmax X-ray Diffraction, RINT-2200 with Cu tube for generating CuK α equipped with a Ca and Mg filtered CuK α radiation source ($\lambda = 1.542\text{\AA}$) 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 $5^\circ/\text{min}$. Then, XRD pattern would be used to characterize.