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
- Alcohol ethoxylate (EO9) 99.95% purity was supplied by Thai-Ethoxylate
- Calcium hydroxide 99.95% purity was purchased from Sigma-Aldrich
- Deionized water
- Dimethyldodecylamine oxide (DDAO) 99% purity was purchased from Sigma-Aldrich
- Ethylenediamine disuccinic acid (EDDS) was purchased from Sigma-Aldrich
- Hydrochloric acid was obtained from Lab Scan
- Magnesium chloride 99% purity was purchased from Sigma-Aldrich
- Methyl ester sulfonate (MES) was provided by PTT-Chemical
- Sodium hydroxide was obtained from J.T. Baker
- Stearic acid 98.5% purity was purchased from Sigma-Aldrich

3.2 Equipment

- 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)

3.3 Methodology

3.3.1 Soap Scum Preparation

Calcium and magnesium stearates are the models of soap scum used in the experiments. For calcium stearate, it was synthesized from the reaction between calcium hydroxide and stearic acid as a ratio of 1:1. The calcium hydroxide was dissolved in deionized water while the stearic acid was dissolved in ethanol. Then, the stearic acid solution was mixed with the calcium hydroxide solution. After that, soap scum was formed as white solid particles. The solution was left for a day to reach equilibrium. Then, the solution was filtered by using a 0.2 micron nylon membrane and follows by rinsing 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 h. For the magnesium stearate, the synthesis procedure was similar to that for calcium stearate except magnesium chloride to be used instead of calcium hydroxide.

3.3.2 Solubility Experiments

For the equilibrium solubility of calcium and magnesium soap scum, the experiments were done by using various types of surfactants; anionic surfactant (MES), nonionic surfactant (EO9) and amphoteric surfactant (DDAO) with different biodegradable chelating agents; GLDA and EDDS. An excess amount of each synthesized soap scum was added in a solution containing 0.1 M of surfactant and 0.1 M of chelating agent. Then, the mixtures were equilibrated for one night. After that, the filtrate samples were taken for analysis of calcium or magnesium using atomic spectrophotometry.

3.3.3 Soap Scum Dissolution Experiments

An excess amount of synthesized calcium or magnesium soap scum was added to a solution containing different surfactant and chelating agent at different equilibrium solution pH values. After that the solution was heated to around 70°C in a water bath for 3 h. Next, all samples were equilibrated at 25°C in the temperature-controlled water bath at least 1 week with routine shaking. After that, the solutions

were filtered using a 0.2 micron nylon filter membrane to separate the remaining undissolved soap scum. Finally, the clear solutions were analyzed by the atomic absorption spectrometer (AAS) (SpectrAA-300, Varian) for the concentration of dissolved calcium or magnesium. The average data were obtained from at least 3 times with less than 1% error.