

CHAPTER III

EXPERIMENTAL

3.1 Materials

(1) Anionic wax emulsion (Mobilcer 67), is a commercial wax emulsion supplied by Mobil Oil Thailand Limited. Typical properties and characteristics are shown in Table 3.1

Table 3.1 The typical properties of anionic wax emulsion

Emulsion Type	Anionic
Wax Type	Intermediate
Wax Melting Point, °C	54
Solids Content, wt %	63
Hard Water Stability, ppm CaCO ₃	100
Viscosity Brookfield, cP	750
pH	9.5

(2) Sodium chloride (NaCl) obtained from APS Ajax Finechem, Australia is analytical purity grade used as an electrolyte.

(3) Aluminium ammonium sulfate ($\text{AlNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$) obtained from Carlo Erba, Italy is analytical purity grade used as an electrolyte.

(4) Water treated by ion exchanger and total hardness as CaCO₃ less than 50 ppm is similar type used in the wax emulsion manufacturing.

- (5) Wet strengthened filter paper (Whatman No. 114) 24 cm diameter obtained from Whatman International Ltd., England.

3.2 Apparatus and Instruments

- (1) pH meter was Schott Gerett model CG 483.
- (2) Moisture analyzer was Sartorius model MA30.
- (3) Inductively coupled plasma atomic emission spectrometer (ICP-AES) for metal analysis was Perkin–Elmer model plasma 400.
- (4) Mixer was Ligthin model 5158.
- (5) Analytical balance was Mettler model AE200 and AT201.
- (6) Instrument for water separability of wax emulsion wastewater was Petrotest (ASTM D1401-96), consisted: 100 ml cylinder graduated from 5 to 100 ml in 1.0 ml division. Heating bath sufficiently large and deep to permit the immersion of at least two test cylinder in the bath liquid up to the 85-mL graduations and stirring paddle made of stainless steel (length 120 mm, width 19 mm, thickness 1.5 mm).

3.3 Procedure

The experiments were divided into 2 parts; removal of wax emulsion in wastewater by addition of electrolyte and characterization of water quality i.e. pH, COD, oil and grease, suspended solid, total dissolved solid, turbidity, salinity and metal content analysis of aluminium (Al) and sodium (Na).

3.3.1 Phase Separation of Simulated Wastewater of Anionic Wax Emulsion

A simulated wastewater, was prepared from anionic wax emulsion (Mobilcer 67), which a solid content was prepared at 0.3 %, 9.0 %, 3.0 %, 4.9 % and 7.1 % by weight of solid content by dilution anionic wax emulsion with water. The mixed solution was stirred at 300 rpm, for 30 minutes, solid content was analyzed by moisture balance method and reported as shown in Table 3.2.

Table 3.2 The simulated wastewater properties

Sample No.	Solid content, wt %	pH value
SW 1	0.3	7.0
SW-2	0.9	7.6
SW-3	3.0	8.1
SW-4	4.9	8.4
SW-5	7.1	8.8

Solid content is percentage of wax and/or solid particle remaining from heated at a prescribed rate to constant weight, determined by moisture balance method. The solids content is read directly from the balance and moisture is determined by difference.

3.3.1.1 Electrolytes

Two types of electrolyte were used, sodium chloride (NaCl) and aluminium ammonium sulfate ($\text{AlNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$) to demulsify the wastewater emulsion. The electrolyte was varied at 1.0 %, 2.0 %, 3.0 %, 4.0 %, 5.0 %, 6.0 %, 7.0 %, 8.0 %, 9.0% and 10.0 % by weight. The solution temperature was controlled at 25, 30, 40 and 50°C in water bath until the complete phase separation was observed.

3.3.1.2 Stirring Rate and Time for Phase Separation

The simulated wastewater (SW- 4) containing 4.9 % solid content and 7.0 wt % NaCl (or 3.0 wt % when alum was studied) was selected. The stirring rates were varied at 100, 300, 600, 900, and 1,200 rpm while the temperature at each rate was kept constant at 25, 30, 40 and 50°C. The volumes of water and wax phases were observed and recorded every 10 minute until the phase volumes were no longer changed.

3.3.1.3 Actual Wastewater of Wax Emulsion

The optimum conditions obtained from the simulated wastewater were selected for actual wastewater study. Actual wastewater was taken from flushing process after anionic wax emulsion production.

3.3.2 Study of Water Quality

3.3.2.1 pH Measurement [17]

pH of solution was determined by using electrometric method (4500-H⁺B) which the electrometric pH measurement is determination of the activity of the hydrogen ions by potentiometry using a standard hydrogen electrode and a reference electrode.

3.3.2.2 Chemical Oxygen Demand (5220B) [17]

Most types of organic matter are oxidized by a boiling mixture of chromic and sulfuric acid. A sample is refluxed in a strongly acid solution with a known excess of potassium dichromate ($K_2Cr_2O_7$). After digestion, the remaining unreduced $K_2Cr_2O_7$ is titrated with ferrous ammonium sulfate to determine the amount of $K_2Cr_2O_7$ consumed and oxidizable organic matter is calculated in a term of oxygen equivalent.

3.3.2.3 Oil and Grease (5520B) [17]

Oil and grease was analyzed by partition-gravimetric method and a dissolved or emulsified oil and grease was extracted from water by intimate contact with an extracting solvent, trichlorotrifluoroethane or a mixture of 80 % *n*-hexane and 20 % methyl- *tert*-butyl ether. Some extractable matters especially unsaturated fat and fatty acid which are oxidized readily required special precautions regarding to temperature and solvent vapor displacement to minimize the effect. After oil and grease is extracted from the sample, the solvent is recovered by distilling in a tarred distilling flask until

the organic solvent is free of residue and the weight of oil and grease is determined from the weight gained in the tarred distilling flask.

3.3.2.4 Suspended Solid (2540B) [17]

A well mixed sample is filtered through a weighed standard glass-fiber filter and the residual retained on the filter is dried to a constant weight at 103 to 105°C. The increase in weight of the filter represents total suspended solid.

3.3.2.5 Total Dissolved Solid (2540C) [17]

A well mixed sample is filtered through a weighed standard glass-fiber filter, and the filtrate is evaporated to dryness in a weighed dish and dried to constant weight at 180°C. The increase in dish weight represents the total dissolved solid.

3.3.2.6 Turbidity (2130B) [17]

The turbidity of samples was measured by turbidimeter consisting of a nephelometer with a light source for illuminating the sample and a photoelectric detector with a readout device to indicate intensity of light scattered at 90° to the path of incident light.

3.3.2.7 Salinity (2520B) [17]

The determination of salinity used indirect method involving the measurement of conductivity (2510B), which used a conductivity bridge calibrated with standard seawater (equal to a KCl solution containing a mass of 32.4356 g in a mass of in a 1 kg of solution). A calibration curve of standard solutions is required for salinity calculation of sample solutions from conductivity value.

3.3.2.8 Metal Analysis (ASTM D5185)

Metal content i.e., aluminium (Al) and sodium (Na) in solid wax particles and clear water phase were analyzed by ICP method. Wavelength selection, Al was analyzed at wave length 396 nm and Na was analyzed at wave length 588 nm.

Preparation of Test specimens and Standards

- 1) Blank prepared by diluting the base oil ten-fold by mass with kerosine.
- 2) Working Standard prepared by weigh 2 g of 0.0500 mass % single element organometallic standards in 100 ml glass bottle, add 8 g of base oil and 90 g of kerosine.
- 3) Test Specimens prepared by weigh 1 g of solid wax particle sample into 50 ml volumetric flask, add 9 g of base oil and 40 g of kerosine. The mixed solution kept until a solid wax particle was dissolved.

Operating parameters for metal content in solid wax particles

- 1) Argon Flow (75-85 psi)

Plasma	15 L/min
Auxiliary	2.0 L/min
- 2) Nebulizer Meinhard Type K, concentric with recessed capillary tip.

Flow	1.5 L/min
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- 3) Injector Stepped bore, 4 mm i.d. to 1.2 mm i.d., quartz.
- 4) Torch Perkin-Elmer corrosion-resistant demountable torch
- 5) RF Power Incident, 1100 watts nominal.
- 6) Viewing height 15 mm
- 7) Sample uptake rate 1 mL/min

