

CHAPTER III

EXPERIMENTAL

2.1 Materials

High-density polyethylene (HDPE) sheet samples were obtained from commercial drinking water blow-molded bottles produced by SVB Drinking Water Company (Thailand). Printing of these bottles was done at CK-Plastic Company (Thailand). Prior to printing, the HDPE bottles were flame-treated. A blue solvent-based ink formulation (UPE-B4009/2) from Uni Ink Company (Thailand) was screen-printed on the treated HDPE surfaces. The printed part of the bottles were then cut into 8 mm × 40 mm sheets.

N-dodecyltrimethylammonium bromide (DTAB) (99% purity; powder) and n-tetradecyltrimethylammonium bromide (TTAB) (98% purity; powder) were purchased from Aldrich (USA), while n-hexadecyltrimethylammonium bromide (CTAB) (98% purity; powder) was purchased from Fluka (Switzerland). Sodium chloride (NaCl) was purchased from Aldrich (USA). Sodium hydroxide (NaOH) (98% purity), purchased from EKA Noble (Sweden), and hydrochloric acid (HCl) (AR grade), purchased from BHD (Germany), were used for pH adjustment. All of these chemicals were used as-received.

Ink powder was prepared by mixing the as-received ink with an appropriate solvent and the mixture was evenly pasted onto a 15" × 10" glass plate. The layer of ink was made as thin as possible to hasten the drying. The painted glass was later dried in an oven at 60°C over night, after which time the ink was scraped off from the glass surface, ground in a mortar, and finally sieved into powder of about 100 mesh or less (i.e., <150 μm). The as-prepared ink powder was kept in a desiccator prior to further use. The chemical composition of the ink powder was investigated by a Fourier-transformed infrared spectrometer (FT-IR; Bruker Vector 3.0), operating at a resolution of 4 cm⁻¹ and a frequency range of 4000 to 400 cm⁻¹.

2.2 Critical Micelle Concentration Measurement

At $30 \pm 1^\circ\text{C}$, the critical micelle concentration (CMC) of pure DTAB, TTAB, and CTAB solutions was determined based on conductivity, using a conductivity meter (Orion, 125). Surface tension values, used to determine the CMC of the CnTAB solutions in the presence of 0.1 M NaCl, were measured by a drop shape analyzer (KRÜSS, DSA10-Mk2). The temperature was maintained at $30 \pm 1^\circ\text{C}$. For both surface tension and conductivity, the CMC is the surfactant concentration at which there is an abrupt change in the slope of the property as a function of the surfactant concentration.

2.3 Viscosity Measurement

The viscosity of each CnTAB solution was measured in comparison with that of water by a Ubbelode viscometer. The Ubbelode tube containing a sample solution was partially submerged in water, the temperature of which was equilibrated to $30 \pm 1^\circ\text{C}$. Prior to each measurement, the sample solution was filled in the Ubbelode tube and was left for temperature equilibration for 15 minutes. During measurement, the solution was then raised up and then allowed to flow gravitationally. The elapsed time for the solution to flow passing two marked lines was recorded. The measurement was repeated five times, from which an average value was calculated. The viscosity of the solution is assumed to relate to that of water, according to the following relationship [14]:

$$\frac{\eta_1}{\eta_0} = \frac{\rho_1 t_1}{\rho_0 t_0}, \quad (2)$$

where η is viscosity, ρ is density, and t is the measured elapsed time. “1” represents the measured CnTAB solution and “0” represents water.

2.4 Zeta Potential Measurement

A very small amount of the as-prepared ink powder was added into a CnTAB solution. The pH level of the mixture was adjusted to either 11 or 12 and the mixture was stirred for 24 hours. The temperature of the mixture was equilibrated at $30 \pm 1^\circ\text{C}$. The as-prepared mixture was then transferred to an electrophoretic cell of a zeta meter (Zeta Meter, 3+), equipped with a microscope module. After applying a suitable voltage according to the solution conductivity, the time for any visible ink particle to move for a certain distance was measured. For a given data set, at least 20 ink particles were monitored, from which the average time was calculated. The average time was then used to calculate the average zeta potential value for that particular data set.

2.5 Solubilization Measurement

The concentration of solubilized ink in CnTAB solutions with or without the presence of 0.1 M NaCl was indicated by first mixing 10 mg of the as-prepared ink powder in a 15 mL CnTAB solution at pH 12 in a 50 mL Erlenmeyer flask. The flask was then placed in a shaking water bath with the temperature of the water being varied between 30 and 45°C and the shaking frequency of the sample stage being set at 200 cycles/min for 4 hours. The solution was later filtered to remove undissolved ink particles using filter paper (Whatman no. 7402-001; average pore size = $0.2 \mu\text{m}$). The UV absorbance of the filtrate was measured by a UV-visible spectrophotometer (Shimadzu, UV-2550) at a wavelength of 273.8 nm. The absorbance for accurate measurement is in the range of 0.1 to 0.8. If the sample solution showed an absorbance greater than this range, it was further diluted. The absorbance versus the concentration of CnTAB solutions is a qualitative measure for ink binder solubilization in the CnTAB solutions as increased absorbance corresponds to increased solubility of the binder in the surfactant solutions due to increased solubilization in micelles.

2.6 Deinking Experiments

To investigate the effects of alkyl chain length, surfactant concentration, pH, temperature, and salinity on deinking efficiency, DTAB, TTAB, and CTAB solutions were prepared at different concentrations, pH levels (i.e., between 11 and 12), temperatures (i.e., between 30 and 45°C), and salinity (i.e., with or without 0.1 M NaCl). The as-prepared printed HDPE specimens were first pre-soaked (without shaking) in a 15 mL surfactant solution for 2 hours and further soaked while being shaken at 200 cycles/min for another 2 hours in a shaking water bath. Then, the specimens were washed with deionized water and later dried in open air at room temperature overnight. The amount of ink on the plastic before and after deinking was measured using the optical scanning method (Songsiri, 2002). In this method, each plastic specimen was carefully positioned on and scanned by a HP ScanJet 4C optical scanner using the factory settings. The scanned files were analyzed by a copy of Adobe Photoshop 5.5 by counting the number of pixels (proportional to the amount of ink present) on the plastic surface. The amount of ink removed (%) was then calculated based on the following relationship (Songsiri, 2002):

$$\text{Ink removed (\%)} = \left[\frac{\text{pixels}_{\text{before deinking}} - \text{pixels}_{\text{after deinking}}}{\text{pixels}_{\text{before deinking}}} \right] \times 100. \quad (1)$$