CHAPTER III

METHODOLOGY

3.1 Materials

3.1.1 Solvent-based Ink Printed HDPE Plastic Containers

Plastic containers tested for this research were totally composed of high-density polyethylene and printed with blue color solvent-based ink by the screen printing process. Prior to printing, the containers were flame-treated in order for the ink to adhere well to the surface. All the tested containers, which were treated and printed by the company, were purchased from SVB Drinking Water Company.

3.1.2 Surfactants

The cationic surfactant, n-hexadecyltrimethylammonium bromide (CTAB), with 98% purity in the powder form was supplied by the Fluka Company, Switzerland. The anionic surfactant, sodium dodecyl sulfate (SDS), was in powder form with 96.5% purity and obtained from Henkel Company. The nonionic surfactant, polydisperse nonylphenol polyethoxylate with an average degree of polymerization of 10 ($NP(EO)_{10}$), in viscous liquid form was supplied by the Lion Company. All surfactants were used as received without further purification. The chemical structure, properties and solution concentrations of all surfactants are shown in Tables 3.1and 3.2.

Table 3.1 Properties of Surfactants Used for Deinking Experiments

Surfactant	Chemical structure	Molecular weight(g/g-mole)	CMC (mM)
CTAB	$C_{16}H_{33}{}^{+}N(CH_3)Br^{-}$	364.46	0.92
SDS	$C_{12}\ H_{25}SO^{-}{}_4Na^{+}$	288.38	8.2
$NP(EO)_{10}$	$C_9H_{19}(C_6H_4)O(C_2H_4O)_{10}H$	660	4.2×10^{-2}

Table 3.2 Surfactant Concentrations Used for Deinking Experiments

Surfactant	At CMC	Below CMC	Above CMC
CTAB	0.92mM (0.03 w%)	0.6 mM (0.02 w%)	30 mM (1 w%)
SDS	8.2mM (0.2 w%)	5 mM (0.1 w%)	35 mM (1 w%)
$NP(EO)_{10}$	4.2×10^{-2} mM (0.0027 w%)	0.03 mM (0.002 w%)	15 mM (1 w%)

3.1.3 Abrasive

Small irregular shape ceramic pieces were used as the abrasive in order to facilitate the detachment of loosened ink from the rigid plastic surfaces.

3.1.4 pH Adjusting Chemicals

Sodium hydroxide with 98% purity was received from EKA Nobel Company and 37% hydrochloric acid was supplied by Merck Company.

3.1.5 Water

Triple distilled water was used in all deinking experiments.

3.2 Experimental Procedures

3.2.1 Analysis before Deinking Process

Several experiments were conducted to evaluate the effect of surfactant on deinking of HDPE bottles under various conditions. The amount of ink on each sample was determined by using Attenuated Total Reflection Fourier Transform Infrared (ATR-FTIR) Spectroscopy (Bruker RFS 100) and an optical scanning method.

A plastic sample containing blue-colored printing ink (approximately 1 cm × 4 cm) was cut from the printed area of the plastic bottle and weighed. The transmission spectra of printed, nonprinted, and deinked (partially and totally) plastic samples were measured by ATR-FTIR spectroscopy. This technique was used as a surface analytical technique and has been applied to examine the surface properties of all plastic samples. Each representative sample was closely placed on a ZeSe crystal with 45° angle of incidence that

reflect IR radiation multiple times off the sample plastic surface. The resulting spectra of both nonprinted and printed plastic samples were used as a standard for comparison with deinked plastic samples.

For optical scanning, a HP LaserJet 4c scanner and Adobe PhotoShop program were used to analyse the selected ink regions using a histogram method. Each representative plastic sample was scanned at a specified position and conditions. In order to prevent the reflection from the white surface of the scanner cover, a black poster board was placed behind the samples during scanning. After scanning, image files were imported into an Adobe PhotoShop program to quantify the amount of ink by a histogram analysis.

3.2.2 Experimental

All surfactant solutions were prepared with triple-distilled water at room temperature and stirred with a magnetic stirrer to produce homogeneous solutions. CTAB solution had to be warmed up slightly, as it is not dissolved in water at room temperature. After that, the pH level of the surfactant solutions were adjusted immediately by adding hydrochloric acid or sodium hydroxide. Nichiryo Autoclavable Model 5000DG digital micropipettes 100 and 1000 were used to define the amount of acid or base to obtain the required pH level. All pH measurements were done with a pH meter (Benchtop pH/ISE Meter, Model 420A with Triode pH electrode Model 91-578N). After analysing the samples (before deinking) by ATR-FTIR spectroscopy and optical scanning method, each sample was placed in 15 mL surfactant solution in a conical flask and allowed to soak with and without 25 irregular-shaped ceramic pieces for varying periods of time. The flasks were then placed in a GFL 1086 model shaking water bath and allowed to shake for a pre-determined period of time at a speed of 260 oscillations per minute. After shaking, all plastic samples were removed from the flasks, washed several

times with distilled water and dried in a Gallenkamp vacuum oven at 50°C overnight. All experiments were conducted at room temperature and each experiment was repeated at least twice.

3.2.3 Analysis after Deinking Process

The degree of deinking for all deinked samples were first evaluated visually and then reweighed. After reweighing, the deinked plastic samples were rescanned on the HP LaserJet scanner using the same procedures as for the original samples and the amount of ink removal determined.

The IR spectra for all deinked plastic samples were also retaken by ATR-FTIR spectroscopy to qualify ink removal using the same procedures as for the original samples. This technique could be used to analyse approximately the top 1 micron of the sample plastic surface. Comparing the spectra taken from the nonprinted and deinked plastic samples before and after deinking experiments, it was found that the degree of deinking can be determined qualitatively by this technique.