



CHAPTER III EXPERIMENTAL

3.1 Materials

Methyl acrylate monomer (99% purity, MA, $C_4H_6O_2$) was purchased from Fluka. 2,2'-Azobisisobutyronitrile (98% purity, AIBN, $C_8H_{12}N_4$), a water insoluble initiator, and Dodecylbenzenesulfonic acid, sodium salt (DBSA, $C_{18}H_{29}SO_3Na$), an anionic surfactant, were purchased from Aldrich. Sodium chloride (NaCl) was obtained from Carlo Erba. Absolute ethyl alcohol and toluene (A.R. grade) were purchased from Lab Scan. Concentrated hydrochloric acid and sodium hydroxide (37%, J.T. Baker Inc., USA and Carlo Erba, Italy, respectively) were used for the adjustment of pH.

Polyester fabric was washed several times in a washing machine using hot water until it was free from any remaining lubricants and finishes prior to use. Mixed cement was purchased from Siam Mortar Co., Ltd. Deionized water was used in all experimental work.

3.2 Equipments

3.2.1 UV-Spectrophotometer

The amounts of DBSA and methyl acrylate in the supernatant were measured using Shimadzu, Model 2550 UV-spectrophotometer at wavelength 224 and 238 nm, respectively.

3.2.2 Fourier Transform Infrared Spectroscopy (FT-IR)

The FT-IR spectra of poly(methyl acrylate) which extracted from treated polyester fabric was obtained using a Nicolet Nexus 670 FT-IR spectrometer in the frequency range of $4000-400\text{ cm}^{-1}$ at a resolution of 4 cm^{-1} to confirm the formation of a polymeric thin film on the fabric surface. The thin film of poly(methyl acrylate) on polyester fabric was extracted by using boiled toluene. After the solvent was evaporated off, the remaining polymer was analyzed and KBr pellet technique was applied in the preparation of powder samples.

3.2.3 Scanning Electron Microscope (SEM)

Surface morphology of treated polyester fabric was studied by JOEL 5200-2AE scanning electron microscope to observe the uniformity and coverage of the film. The polyester fabrics were sampled randomly from different positions and sputter coated with gold under vacuum before observation to make them electrically conductive.

3.2.4 Contact Angle Measurement

Static contact angles with water were measured for both sides of the fabric surface using sessile drop method on a DSA10-Mk2 contact angle measuring instrument. Measurements were carried out at 30°C. For each sample, five drops of liquid are placed in different parts of the sample on both sides. A drop of test liquid was introduced by injecting exactly 10- μ L of test liquid with a 10- μ L syringe. The average value of all measurements was then calculated.

3.2.5 Flexural Sample Preparation and Experiment Procedure

Wet cement mortar was poured into a mold for a half and then the untreated or treated polyester fabric was introduced onto the wet cement. After that, another half portion of wet cement was poured to cover another side of the polyester fabric. The samples were allowed to harden in air for 4-5 day, after which, the flexural tests were carried out using Instron Universal Testing Machine model 4206.

3.3 Methodology

3.3.1 Determination of Equilibrium Adsorption Time

The solution of DBSA of a desired concentration and pH ([DBSA] = 5mM, pH less than the pH at PZC). A 25 ml aliquot of the solution was pipetted into a 32 ml vial containing 0.5 g of polyester fabric. The sealed vial was placed in a shaker bath at 30°C for a set time. Small amount of supernatant was sampled at various times to determine the equilibrium adsorption time of DBSA on polyester fabric. The concentration of DBSA in solution was determined by Shimadzu UV-spectrophotometer model 2550. The adsorbed DBSA on polyester fabric was calculated by taking the difference between the initial and final concentration of

DBSA. A plot of time versus adsorbed surfactant was carried out to determine the time for equilibrium adsorption when there was no longer change in adsorbed surfactant. The wavelength of maximum adsorption for aqueous solution of DBSA was investigated from the calibration curve.

3.3.2 Determination of Surfactant Adsorption Isotherm

The adsorption isotherm of DBSA on polyester fabric with and without electrolyte was obtained by exposing 0.4g of polyester fabric to 25 ml of DBSA solution of known initial concentration. The mixture was equilibrated at 30°C for optimum time in a sealed vial. The amount of DBSA in supernatant was measured by a UV spectrometer. The initial DBSA concentration in this experiment was varied from 10 to 6,000 μM , which covered the region below and above the CMC of DBSA. A plot of equilibrium supernatant concentration versus the amount of adsorbed surfactant yielded, which is known as the surfactant adsorption isotherm was conducted.

3.3.3 Determination of Monomer Adsolubilization Isotherm

Stock methyl acrylate-in-surfactant and surfactant solution were used to prepared samples containing constant DBSA concentration with different methyl acrylate concentration. The methyl acrylate/DBSA solution of various ratios was added to 0.4g of polyester fabric in 32 ml vials sealed with paraffin. The mixture was equilibrated at 30°C for optimum time in a shaker bath. The methyl acrylate concentrations before and after equilibrium were measured by Shimadzu UV-spectrophotometer model 2550. Adsolubilization of methyl acrylate was calculated by simple mass balance. The adsolubilization isotherm was plot between adsolubilization of methyl acrylate on polyester fabric ($\mu\text{mol/g}$) and equilibrium concentration of methyl acrylate solution (M).

3.3.4 Admicellar Polymerization

Polymerization of methyl acrylate on polyester fabric was carried out using optimum DBSA concentration and pH with the desired amount of NaCl, DBSA : monomer molar ratio, initiator : monomer molar ratio. A $5 \times 5 \text{ cm}^2$ polyester fabric was placed in a 32 vial containing 30 ml of the DBSA solution. The sealed vial was placed in a shaker bath at 30°C for optimum time to reach equilibrium. Then the temperature was raised to 75°C to initiate the polymerization reaction. After the

polymerization, the reaction will be quenched by immersion the vials in an iced bath. The fabrics were removed from the vials and washed 3 times in distilled water at 80°C for 30 min to remove the upper layer of DBSA and expose the polymer film. Finally, the fabrics were placed in an oven 65°C overnight to remove any remaining water and residual non-polymerized monomer.

3.3.5 Characterization of The Treated Fabric

3.3.5.1 *Identification of The Coated Film*

The presence of poly(methyl acrylate) film was confirmed by using a Nicolet Nexus 670 FT-IR spectrometer in the frequency range of 4000-400 cm^{-1} at a resolution of 4 cm^{-1} . The thin film of poly(methyl acrylate) on polyester fabric was extracted by using boiled toluene. After the solvent was evaporated off, the remaining polymer was analyzed and KBr pellet technique was applied in the preparation of powder samples.

3.3.5.2 *Surface Morphology of The Treated Fabric*

The morphology of the treated fabric surface was studied by Scanning Electron Microscope JOEL, Model JSM 5200 to observe the uniformity and coverage of the film. The polyester fabrics were sampled randomly from different positions and sputter coated with gold under vacuum prior to observation to make them electrically conductive. Magnification used was x3500.

3.3.6 Testing

3.3.6.1 *Contact Angle Measurement*

Static contact angles with water were measured for both sides of the fabric surface using sessile drop method on a DSA10-Mk2 contact angle measuring instrument. Measurements were carried out at 30°C. For each sample, five drops of liquid are placed in different parts of the sample on both sides. A drop of test liquid was introduced by injecting exactly 10- μL of test liquid with a 10- μL syringe. Contact angle was measured after the drop is put down from the syringe every second for 30 second using Tacker man function. The average value of all measurements was then calculated.

3.3.6.2 Flexural Test (Sample Preparation and Experiment Procedure)

Wet cement mortar was poured into a mold for a half and then the untreated or treated polyester fabric was introduced onto the wet cement. After that, another half portion of wet cement was poured to cover another side of the polyester fabric (see Figure 3.1). The samples were allowed to harden in air for a week, after which, the flexural tests were carried out using Instron Universal Testing Machine model 5567. The specimen dimension is 100 mm in width, 100 mm in length, and 5 mm in depth. The test conditions are 1 KN Load cell, 80 mm support span, and at rate of crosshead motion 0.5 mm/min. Figure 3.2 shows the flexural test configuration.

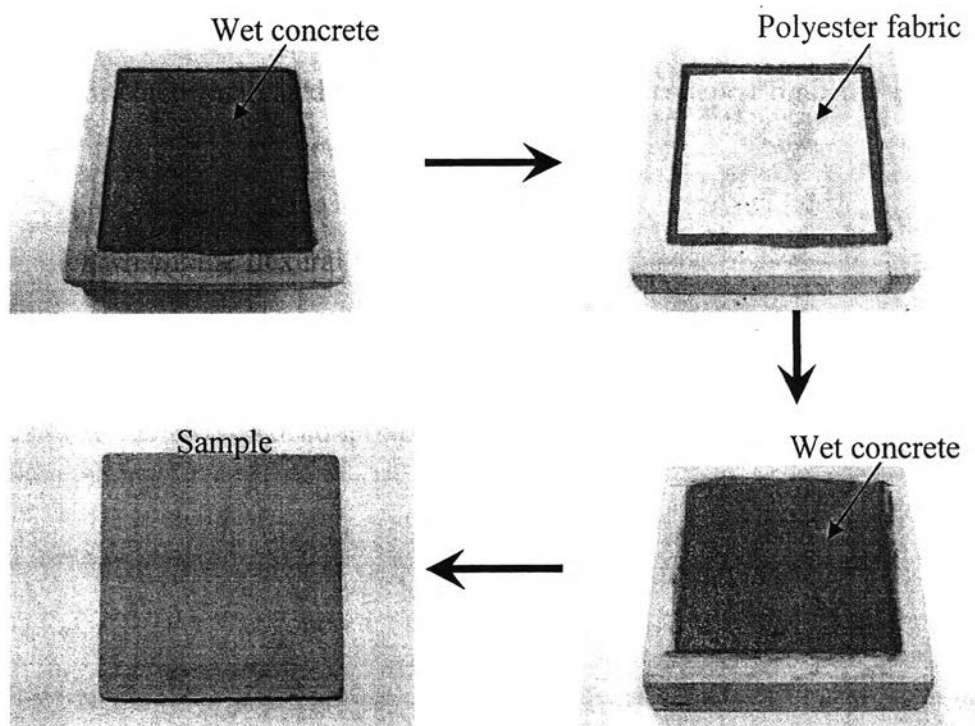


Figure 3.1 Flexural sample preparation.

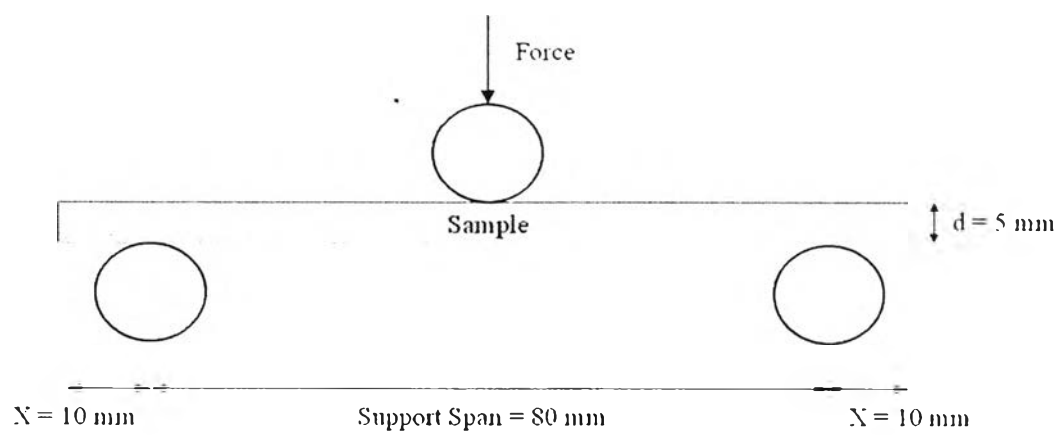


Figure 3.2 The flexural test configuration.