CHAPTER III
EXPERIMENTAL SECTION

3.1 Materials

-Cotton fabric was ordered from Boon Chauy Co. Ltd.

-Linear alkyl benzene sulfonate (LAS) was provided by Unilever Thai Holding Ltd. Carbon chain length is between 8-12 atoms and average molecular weight is 344.

-Sodium persulfate, hydrochloric acid, and sodium chloride were purchased from BHD Laboratory Supplies, Merck, Ajex Chemicals, respectively.

-3,4Dichloro-1-butene was purchased from Aldrich Chemicals Company.

-4-Chloromethyl styrene and sodium styrene sulfonate were purchased from Fluka Chemicals Ltd.

All chemicals were used as received except for 4-chloromethyl-styrene.

3.2 Instruments

-Oven (Memmert)

-Shaker bath GFL 1086

-SEM Joel, model JSM-5200

-TGA, Dupont TGA 2590+

-UV spectrometer Cecil CE2040

-pH meter Orion, model 420A

-Color-eye7000 spectrophotometer (Macbeth)

3.3 Methodology

3.3.1 Preparation of Cotton Fabric

The cotton fabric used in the experiment is a plain weave fabric with a fabric weight of 150 g/m^2. The fabric was washed several times in water at 90°C to remove any residual surfactant from the bleaching process. UV detector was used to detect the amount of surfactant in washed liquid at 225 nm. An absorbance below 0.08 was judged acceptable.

3.3.2 Preparation of Monomer

- 4-Chloromethyl styrene

4-Chloromethyl styrene or 4-vinyl benzyl chloride is a styrene derivative. The technique for inhibitor removal was as described by Edward et al (1973). 10% wt NaOH solution and the monomer were added into a separatory funnel. The mixed solution was then shaken and left to stand until the solutions separated. The lower orange phase containing the inhibitor was discarded and the process was repeated until a clear solution in the lower phase was obtained. Finally, the amount of NaOH in the pure monomer was removed by washing with distilled water until the monomer has a neutral pH as checked by a pH meter.

- 3,4-Dichloro-1-butene

3,4-Dichloro-1-butene was used as received.

- Sodium styrene sulfonate

Sodium styrene sulfonate was used as received.

3.3.3 Admicellar Polymerization

The required concentration of LAS solution was prepared from a stock solution of 50,000 μM LAS. The pH of the LAS solution was adjusted to 4 by addition of 1 M hydrochloric acid. NaCl was added in the cases of 3,4-dichloro-1-butene and 4-chloromethyl styrene. 20 mL of the prepared LAS

solution was pipetted into a 24 mL screw cap vial. The monomer was injected into the solution to give a LAS : monomer ratio ranging from 1:1 to 1:15. A piece of 0.5g cotton cut into a size of 5.5x5.5 cm^2 was added to the solution. The vial was then sealed with aluminum foil and the cap was screwed in. The vials were maintained in the shaker bath at 30°C for 15 hrs. After that the predetermined quantity of 2 M $Na_2S_2O_8$ was injected into the vial. The polymerization was carried out at 80°C for 6 hrs. After polymerization the vial was water-cooled and the treated cotton was removed.

3.3.4 Washing Procedure

The treated cotton was washed by stirring in water at room temperature one time. Next, the cotton was washed with distilled water at 80 °C at least 5 times. The ratio of treated fabric to distilled water was 0.5g : 50mL. The washing time was 15 minutes. The residual surfactant in the washed liquid was detected by UV detector at 225 nm. The acceptable absorbance for LAS was below 0.08. Finally, the treated cotton was dried in the oven at 110°C for 6 hrs and was kept in the desiccator before taken out for property testing.

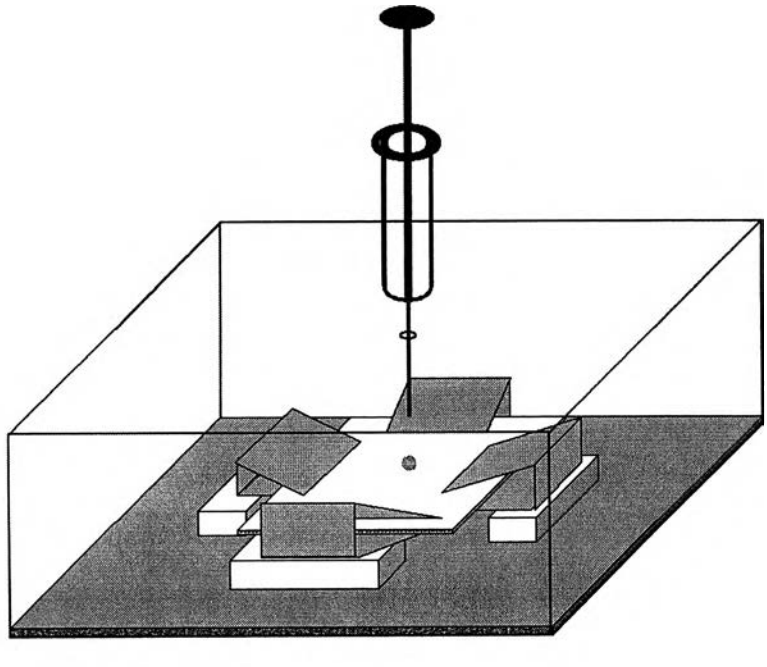

Figure 3.1 Dropped test method.

3.3.5 Hydrophobic Test

The treated cotton was tested for its hydrophobic property by dropped test method. The fabric was stretched on four sides by four clips. Next, 10 μL of distilled water was injected by syringe to form a droplet on the fabric surface as shown in Figure3.1. The time taken for the droplet to disappear was determined.

3.3.6 Dyeing Test

The dye solution was prepared by using 1%wt of dye based on weight of cotton. 20 ml of the dye solution was then pipetted into the vial. The treated cotton was submerged in dye solution for 1 hour at 30°C with shaking in the shaker bath. After dyeing was finished, the dyed cotton was taken out and washed with distilled water four times at 30°C. Finally, the cleaned cotton was dried in the atmosphere and the color strength was observed.

3.3.7 Color Strength Analysis

The color strength of the dyed fabric was determined by spectrophotometer. The color strength or K/S is a dimensionless parameter which is used to determine the amount of colorant in the material. This value can be calculated from the reflectance of colored sample by spectrophotometer. The reflectance of sample was observed in the visible region of spectrum from 400 to 700nm and lambda that gave the lowest reflectance was used to calculate the color strength of dyed fabric by Kubelka-Munk equation x(See Equation 3.1).

$$K/S = \frac{(1-R)^2}{2R} \qquad (3.1)$$

Where K is the absorption coefficient

S is the scattering coefficient

R is the reflectance of the dyed fabric at the wavelength of maximum adsorption (λ_{max})

3.3.8 Washing Fastness Testing

The durability of dye on the treated cotton after dyeing was observed with the washing fastness testing. The washing solution was prepared with 5g of standard soap in 1000mL tapped water. Then 160 times of solution based on the sample weight was added in the container with the cotton sample. Next, the container was placed in the washing machine and temperature was adjusted to 30°C. The washing machine was operated for 30 minutes and washed sample was then taken out. Finally, the sample was cleaned with tapped water for soap removal and was dried before assessing the color change by comparing with gray-scale. There are five grades in washing fastness rating:

5 "Excellence", 4 "Good", 3 "Fair", 2 "Poor", and 1 "Very poor".

3.3.9 Rubbing Fastness Testing

Another method for testing the durability of dyed sample is the rubbing fastness testing. The instrument for rubbing test is shown in Figure 3.2. The test sample was placed under the rubber that was covered with a piece of white cotton. The frequency of rubbing was then adjusted to one round per second for 10 times. After the operation was finished, the piece of cotton was removed from the rubber and the dye stained on the rubbing cotton was compared with gray-scale for staining rating. The rubbing fastness has two methods: dry test and wet test. For dry test, the piece of cotton was dry. But

for the wet test, the piece of cotton was first dipped in the water before it was fixed at the rubber. There are five grades for rubbing fastness:
5 “Excellence”, 4 “Good”, 3 “Fair”, 2 “Poor”, and 1 “Very poor”.

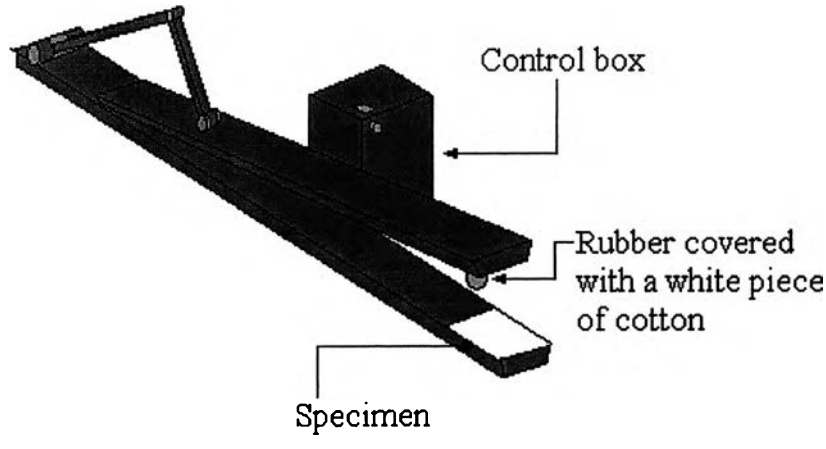

Figure 3.2 The rubbing fastness testing instrument.