

CHAPTER III



Experimental Procedures

3.1 Materials

The fabric used through out this experiment was a mercerized, desized, scoured and bleached 100% cotton.

3.2 Chemicals and Dyestuff

The chemicals used in this project were laboratory reagent grade. An ethanolamine was obtained from MERCK. The crosslinking agent, 1,2,3,4-butanetetracarboxylic acid(BTCA) was obtained from Fluka, and a catalyst, sodium hypophosphite monohydrate($\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$) was purchased from Ajax Chemicals. Sodium hydroxide(NaOH) was provided by J.T. Baker Inc.. Hydrochloric acid was obtained from mallinckrodt Baker, Inc. and commercial reactive dye, Cibacron blue P-B, was kindly provided by CIBA-Geigy.

3.3 Equipment

1. Three-neck round bottom flask
2. Reflux Condenser
3. Mantle Heater
4. Pad Mangle
5. pH meter
6. Minidryer Stenter
7. Ahiba polymat machine
8. Macbeth color-eye 7000 Spectrometer

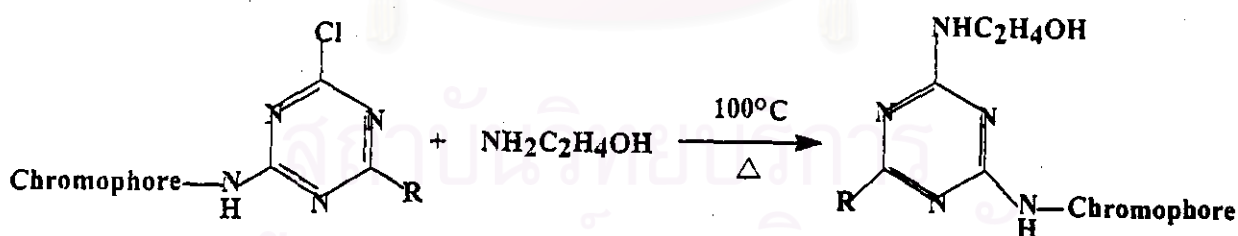
9. Wrinkle Recovery Tester

10. Nicolet Impact 400D FT-IR Spectrometer

11. Glasswares

3.4 The preparation of the hydroxyalkyl dye

To a 250 mL three-necked flask, containing 20 mL of water, 10.0 g of Cibacron blue P-B was added. The aqueous dye solution was stirred and heated on a heating mantle unit. While the solution was raised to the boil, 1 mL of ethanolamine in 10 mL of water was added dropwise. The pH value of the mixture was maintained over 7, given by the presence of excess ethanolamine itself. At the boil, the mixture was then stirred and refluxed for an additional 1 hour. The reaction mixture was then cooled down to room temperature. An isolation of the reaction product was made by adding conc. hydrochloric acid to solify the modified dye. The dye solids were collected by filtration and then dried at 60 °C for 2 hours. The reaction scheme between Cibacron Blue P-B and ethanolamine may be written as follows:



Scheme 3.1 The reaction scheme between Cibacron Blue P-B and ethanolamine to produce an hydroxyalkyl dye.

3.5 Application of the hydroxyalkyl dye to the cotton cellulose in the presence of BTCA as a crosslinking agent

3.5.1 General dyeing procedure

All dyeings were carried out on bleached cotton fabric. Application of dye and other auxiliaries was achieved by padding, pre-drying and curing at temperature ranging from 160 to 180 °C. Padding was performed by immersing cotton fabric in to dye pad-liquor and passing padded fabric through a pair of rollers to squeeze excess liquid and also force dye molecules into the fiber. After being squeezed, padded fabric was pre-dried to remove water at 100 °C for 2 min. and then thermofixed at curing temperature.

3.5.2 Determination of Dye fixation

In order to determine how much the amount of dye that chemically reacted with the cellulose, the thermofixed fabric was divided into two portions, one of which was subjected to a washing-off in the boiled solution containing nonionic detergent (2 g/l) for 15 minutes to remove unfixed dye, then rinsing in tap water and drying at 60 °C. After that, by using an I.C.S. Micromatch colour measurement system (Macbeth 7000), the colour strength of each portion of samples, unwashed and washed fabrics expressed as K/S values were obtained. The values were calculated from reflectance data according to the Kubelka-Munk equation (equation 1):

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

Where K is the absorption coefficient, S is the scattering coefficient, and R is the reflectance at the wavelength of maximum absorption. The higher the K/S value, the greater is the color strength of the fabric.

After washing, a percentage of dye fixation on fabric can be calculated by equation 2 based on equation 1.

$$\% \text{Fixation} = \frac{K/S_a}{K/S_b} \times 100 \quad (2)$$

a = after soaping

b = before soaping

3.6 Studying on the application of the hydroxyalkyl dye to cellulose fabric in the presence of BTCA crosslinking agent

3.6.1 The effect of $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ concentrations on dye fixation

Pad liquors were prepared as follows:

10 g/l Hydroxyalkyl dye

50 g/l BTCA

0,10,20,30,40,50 and 60 g/l $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$

The pH of pad liquor was adjusted to 3 by adding 1.0M NaOH prior to use. Cotton fabrics were padded with prepared pad liquor, padding be performed using a pad mangle set a pressure to give 80% wet pick-up. Padded sample were pre-dried at 100°C for 5 minutes and cured at 180°C for 5 minutes. The degree of dye fixation was carried-out as described in section 4.3.1 .

3.6.2 The effect of time and temperature on the degree of dye fixation

Pad liquors were prepared as follows:

10 g/l	Hydroxyalkyl dye
50 g/l	$\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$
50 g/l	BTCA

The pad liquor was adjusted to pH 3 using 1.0 M NaOH. Cotton fibers was padded with the prepared pad liquor on the pad mangle to give a wet pick-up of 80%. Padded sample was dried at 100°C for 5 minutes. Sample of dried fabric was cut equally into 15 pieces. Each piece was thermofixed at different temperature and time as follows.

160 °C for 1,2,3,4,5 min , respectively

170 °C for 1,2,3,4,5 min , respectively

180 °C for 1,2,3,4,5 min , respectively

and then the degree of dye fixation was measure, as shown in section 4.3.2.

3.6.4 The effect of increasing hydroxyalkyl dye concentrations on the degree of dye fixation

Pad-liquors were prepared as follows:

Pad-liquors			
Dye Conc. (g/l)	BTCA (g/l)	NaH ₂ PO ₂ ·H ₂ O (g/l)	pH
10	50	50	2.5-3.0
20	50	50	2.5-3.0
30	50	50	2.5-3.0
40	50	50	2.5-3.0

Each pad liquor was adjusted to pH 2.5-3 by using 1.0 M NaOH. Fabrics were padded with the prepared pad-liquors, followed by drying and curing as described as earlier. The determination of dye colour yield and dye fixation of each dyed fabric was carried out as usual procedure.

3.7 Analysis of dye-fiber reaction using Fourier Transform Infrared Spectroscopy (Nicolet Impact 400D FT-IR Spectrophotometer)

Infrared spectroscopy is an important tool used for identifying organic dyes, textile fibers, polymers and blends.

3.6.3 The effect of pH on the degree of dye fixation

Pad liquors were prepared with different pH values. A 1.0 M NaOH solution was used to adjust the pH of the solution to 2.0, 2.5, 3.0, 3.5, and 4.0, respectively.

Pad-Liquors			
BTCA (g/l)	NaH ₂ PO ₂ ·H ₂ O (g/l)	Modified dye(g/l)	pH
50	50	10	2.0
50	50	10	2.5
50	50	10	3.0
50	50	10	3.5
50	50	10	4.0

The cotton fabric was impregnated in a pad-liquor, then passed through a pad mangle to give a wet pickup of 80% based on the original weight of the fabric. The treated fabric was dried at 100 °C for 5 minutes, then cured at 180 °C for 5 minutes in a minidryer stenter. The cured fabric was soaped by nonionic detergent at 100 °C for 15 minutes and then dye colour yield was measured, as illustrated in section 4.3.3.

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Principle

Vibrations are a change in the bond dipole moment of molecules. When molecule are stimulated from ground vibrational state to higher vibrational state by interaction with emission of electromagnetic radiation of the appropriate frequency. In phase, the vibrations are enhanced and there is transfer of energy from the incident radiation to the molecule. The spectral transitions are detected by scanning through the frequency, the vibration frequency are shown to be characteristic of particular functional groups in molecules.

The basic components of a fourier transform infrared(FT-IR) spectrometer is illustrated in Figure 3.1

The infrared light source passes into the interferometer which has two mirrors, the transmitted and reflected are combined at the beamsplitter where they produce interference effects. The beam is then passed through the sample and the signal “interferogram” will be generated at the detector. The infrared spectrum is calculated through fourier transformation of this data.

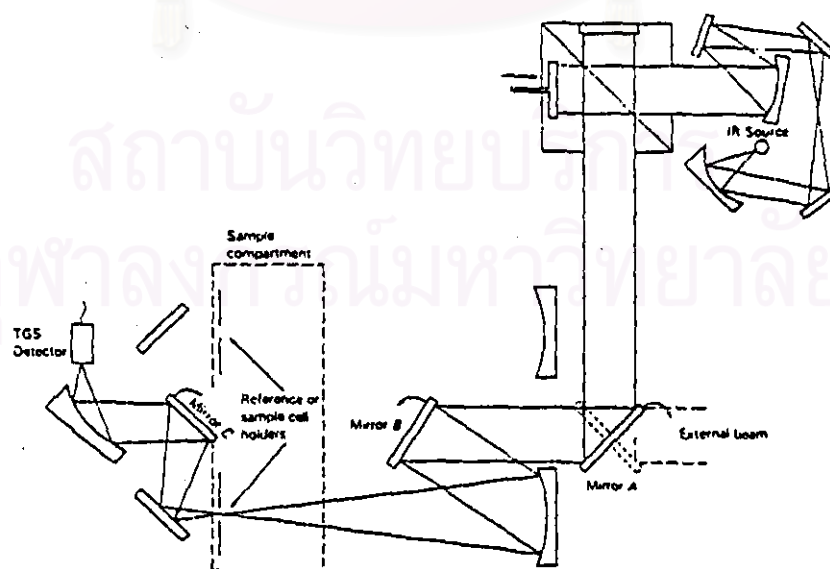


Figure 3.1 The basic components of FT-IR.

The technique for fiber identification is not essentially any different from the examination of other organic compounds. The method of sample preparation used in this experiment is pressed-disc technique. Infrared spectra were obtained on potassium bromide disks of fabric ground by mill. The spectra of the treated fabric were obtained on a Nicolet Impact 400D FT-IR spectrometer. Resolution for all the spectra presented was 4 cm^{-1} . The number of scan was 32. All the spectra were transferred to the computer for further analysis and plotting by Omnic software.

3.8 The evaluation of easy-care properties of dyed fabrics

3.8.1.Recovery Angle Method (AATCC Test Method 66-1984)⁽²⁵⁾

Wrinkle recovery is a property of fabric which enables it to recovery from folding deformation.

Apparatus

Wrinkle recovery tester and accessories, Daiei Kagaku ltd., Kyoto, Japan were used in this experiment.

Preparation of specimens

Based on AATCC Test Method 66-1984, test specimens were taken from samples of fabric that were free from wrinkles. Cut specimens 15x40 mm for testing both directions, parallel to the warp and parallel to the filling, as required. The test specimens were conditioned in the standard testing atmosphere.

Test Procedure

In the long dimension of the specimen, The two edges of it brought together and fold it exactly in half, then placed it between the leaves of a specimen holder. After that, insert the holder and specimen in a plastic press . Put a load of 680 g to the specimen. After 5 min remove the load, transfer a specimen to the clamp on the tester by using tweezers and allow to recover from the crease for 5 min. The crease lined up with a spot at the center of the tester disk and the dangling specimen leg should be lined up immediately with the vertical guide line. Exactly 5 min after the removal of the creasing load, the dangling leg made the final adjustment to the vertical guide line and read the wrinkle recovery value to the nearest degree from the protractor scale by mean of the vernier. Warp and filling way recovery were reported separately in degree.

3.8.2 The evaluation of the strength of dye-fiber bond by washing fastness test

(ISO 105-C04)⁽²⁶⁾

The ISO 105-C04 wash fastness test was used to assess the stability of the carboxylate ester bond. Fastness tests were carried out for dyed fabrics after removing all unfixed dye.

Apparatus

The Ahiba Polymat machine was used in this test.

Preparation of specimen

A specimen measuring 35 mm x 100 mm was attached to a piece of the multifiber adjacent fiber also measuring 35 mm x 100 mm. The composite specimen was placed in the container together, then added 5 g of soap and 2 g of anhydrous sodium carbonate per litre of water by liquor to good ratio 50:1 . treated the composite specimen at 60°C for 30 min. At the end of period, the composite specimen was removed, rinsed in cool water and squeezed it. Opened out the composite specimen and dried it by hanging it in air at room temperature.

The staining of each type of fiber in the multifiber adjacent fabric were assessed with the gray scales.

3.8.3 The determination of the loss of tensile strength of dyed fabrics

(ASTM D5035-95)⁽²⁷⁾

Tensile test was performed on a LLOYD Universal Testing Machine which is type E (constant-rate-of extension). The machine was operated with a leading rate of 300 mm/min, the distance between the clamps was 75 mm, and using 1kN load cell.

ASTM D5035-95 (Strip Method) was used to evaluate strength property of fabrics. Cut each specimen 25 mm plus 20 yarns wide, by at least 150 mm long. The long dimension should be parallel to the direction of testing and force application. Raveled each specimen to give a testing width of 25 mm by removing an approximately equal number of yarns from each side, or 10 yarns from each sides.

Figure 3.2 shows LLOYD Universal Testing Machine.

The tensile property of a specimen was calculated by according to equation 1.

$$\% \text{ Loss strength} = \frac{\text{Strength of standard} - \text{Strength of sample}}{\text{Strength of standard}} \times 100 \quad (1)$$

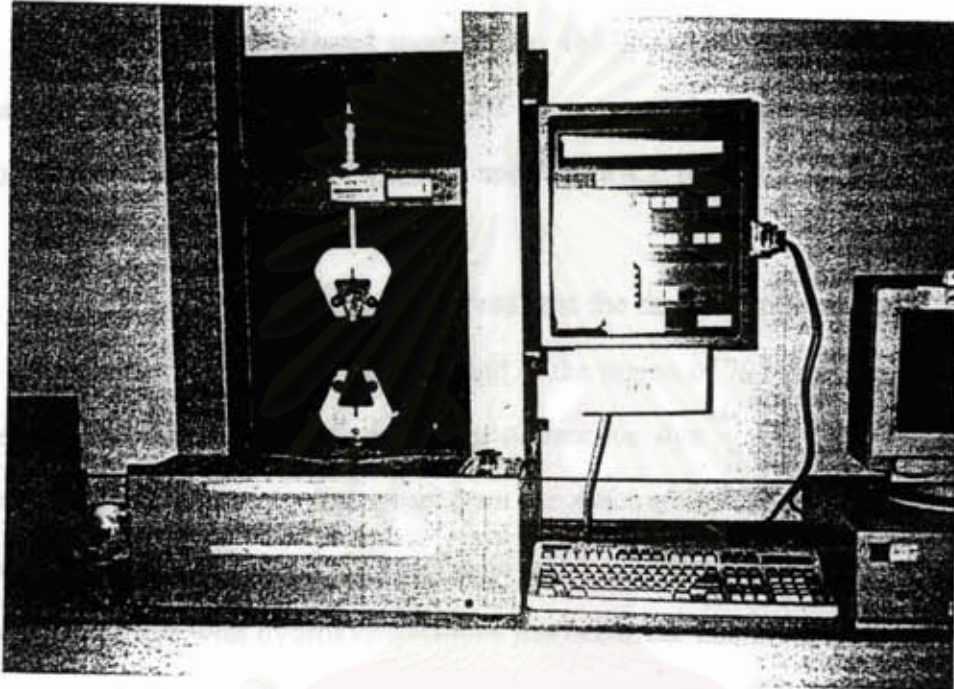


Figure 3.2 LLOYD Universal Machine

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