Chapter III

Experimental

Material

Scoured, plain woven fabric was used throughout this work.

Chemicals and dyes

All chemicals used throughout this work were analytical grade.

Table 3-1 Chemicals used in this project

Chemicals	Company
[3(Methacryloylamino)propyl]trimethyl ammonium chloride (50 wt% solution)	Aldrich Chemical Company, Inc.
Potassium persulphate	May & Baker LTD.
Hydrogen peroxide	Carlo Erba
Sodium hydroxide	BHD Laboratory Supplies
Sodium carbonate	Merck
Sodium chloride	Carlo Erba
Non-ionic detergent	U.N.T. Chemical Co., LTD.
Reactive dyes: Procion Crimson CX-B Modercion Navy Blue Her (C.I.171)	BASF (Red) Modern Dyestuff & Pigments (Blue)

Equipment

1. Rotary dyeing machine & steel pots, Ahiba Polymat®
2. Reflectance spectrophotometer, Macbeth 7000
3. UV/Visible spectrophotometer, Jenway 6405
4. Elemental Analyzer, Perkin Elmer PE2400 series II
5. Optical microscope, Olympus BH2-UMA
6. Xenon Weather Meter, Model X75, Suga Test Instruments Co., LTD. Japan
7. Standard lighting cabinet (Lighting box), VeriVide CAC 60

8. Heating mantle

9. Spatula

10. Glasswears

- Beaker 500, 250, and 100 ml.
- Volumetric flask 100 and 25 ml.
- Pipette 25, 10, and 1 ml.
- Glass rod

3.1 Method of the modification and bleaching of cotton fabric in single-bath process

Application of the modifying agent (MAPTAC) was carried out using exhaustion method.

Firstly, plain woven, scoured cotton fabric was cut into rectangles each of which had a weight of exactly 5.00 grams and was treated in solutions containing different concentrations of MAPTAC ranging from 0-50 g/l in the presence of $K_2S_2O_8$ as an initiator using 150 ml. sealed stainless steel dye pots housed on the Ahiba Polymat® laboratory dyeing machine (Fig. 3.1). The treatment was commenced at different temperatures ranging from 60-90 ^{0} C, at the liquor ratios of 1:10, 1:20, and 1:30 for 45 min. Then 2 ml/L H_2O_2 and 2 g/L NaOH were added to dye pots, and the temperature was raised to 90 ^{0} C. The temperature was maintained for 45 min. before the dye pots being taken out. Finally, the modified bleached cotton fabric was rinsed with water and dried in the open air.

The treatment profile using exhaustion method can be shown as follows:

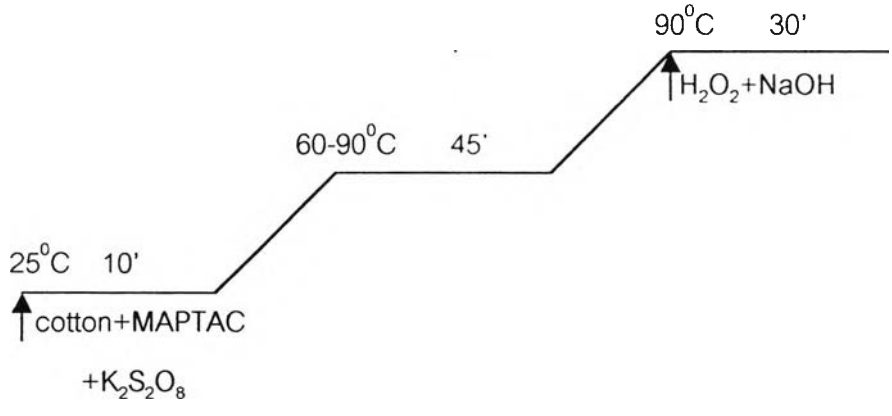

Figure 3.1 The treatment profile of the exhaustion method

3.2 General Dyeing Procedures

3.2.1 Dyeing of modified fabric with reactive dye using exhaustion method

Modified cotton fabrics were dyed in solutions of various concentrations of commercial Procion Crimson CX-Bs and 10 g/L Na_2CO_3 . All dyeings were conducted in 150 ml. sealed stainless steel dye pots housed on the Ahiba Polymat® laboratory dyeing machine at the liquor ratio of 1:10. Dyeing of modified cotton fabric was commenced at 80^0 C for 40 min. The dyed cotton fabrics were taken out and rinsed thoroughly in tap water and cut into two equal portions. One of these portions was soaped in a solution containing 5 g/l NaOH and 5 g/l nonionic surfactant at the boil for 20 min. (liquor ratio 1:50). The dyeing profile of the exhaustion dyeing can be shown as follows:

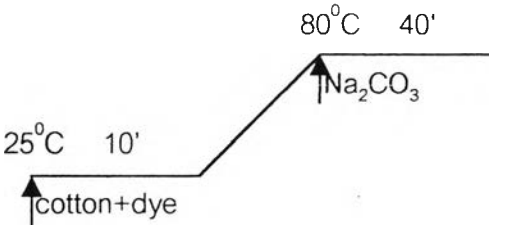

Figure 3.2 The dyeing profile of modified fabric using exhaustion method

In this work, the various factors that affected the dye uptake and the degree of dye fixation were investigated.

3.2.1.1 Effect of increasing concentrations of modifying agent on dye uptake and dye fixation

Concentrations of modifying agent in modifying solutions were varied from 0 to 50 g/l. The treatment was carried out using the method described in section 3.2. The dyeing of modified cotton fabric in the absence of salt was performed in a solution of 2% owf Procion Crimson CX-B at 80^0 C for 40 min. in the presence of 10 g/l Na_2CO_3. The dyeing procedure was followed according to the above method.

3.2.1.2 Effect of increasing concentrations of $K_2S_2O_8$ on dye uptake and dye fixation

Cotton fabrics were treated in solutions containing 20 g/L modifying agent and $K_2S_2O_8$ varied from 2 to 10% based on the modifying agent at 75^0 C for 45 min. Then the modified fabric was dyed in a solution of 2% owf Procion Crimson CX-B at 80^0 C for 40 min. in the presence of 10 g/l Na_2CO_3.

3.2.1.3 Effect of temperature on dye uptake and dye fixation

In this case, cotton fabrics were treated with 20 g/L modifying agent and $K_2S_2O_8$ 4% based on modifying agent at different temperatures ranged from 60 to 90^0 C for 45 min. and then dyed in a solution of 2% owf Procion Crimson CX-B at 80^0 C for 40 min. in the presence of 10 g/l Na_2CO_3.

3.2.1.4 Effect of liquor ratio on dye uptake and dye fixation

The liquor ratio was taken at 1:10, 1:20, and 1:30 respectively with 20 g/L modifying agent and $K_2S_2O_8$ 4% based on modifying agent and then the modified cotton fabrics were dyed in a solution of 2% owf Procion Crimson CX-B at 80^0 C for 40 min. in the presence of 10 g/l Na_2CO_3.

3.2.1.5 Effect of dye concentrations on dye uptake and dye fixation

The dyeing of modified cotton fabric in the absence of salt was investigated. Modified cotton fabrics with different concentrations of modifying agent were dyed in solutions containing various dye concentrations in the range of 1-4% owf in the presence of 10 g/l Na_2CO_3.

3.2.2 Dyeing of unmodified cotton fabric by conventional exhaustion method

Dyeings of unmodified cotton fabrics using different concentrations of commercial Procion Crimson CX-Bs (1%, 2%, 3%, and 4% owf) were conducted in similar manner to the dyeing of modified fabrics. Dyeings of unmodified fabrics were divided into two categories; dyeing with and without electrolyte, NaCl.

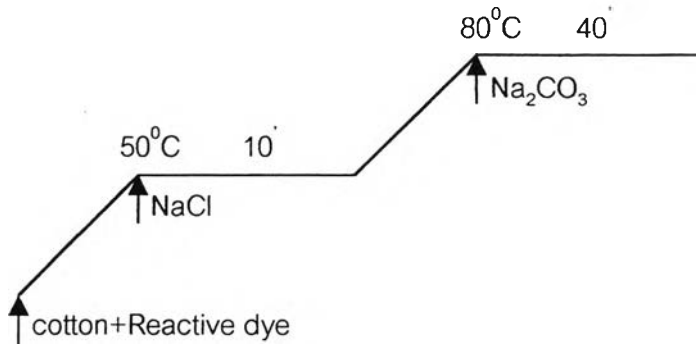

Figure 3.3 The dyeing profile of unmodified fabric using exhaustion method

As indicated in the dyeing profile (Figure 3.3), the various amount of NaCl (20,30,40 g/l) was added into the dye solution at the temperature of 50^0 C for 10 min. to promote dye exhaustion. Then the temperature was raised to 80^0 C and Na_2CO_3 was added to the dye pots. The dyeing was continued for 40 min. After that, the dyed cotton fabrics were taken out and then washed-off in the soap solution of 5 g/l Na_2CO_3 at the boil for 20 min. to remove unfixed dyes. Finally, the washed-off fabric was dried and the degree of dye fixation was measured. The results obtained from the unmodified fabrics were used later in comparison with the modified cotton fabrics.

3.3 Evaluation of whiteness property of modified bleached cotton fabric

Instrumentation has been increasingly used to measure the whiteness of fabrics since it does has the advantage of better precision, i.e. more reliable reproduction of a color measurement than a group of human observers. However, the human eye is still more accurate in determining differences between two samples[34].

To measure the effect of the modifying agent on bleaching performance of modified and unmodified bleached cotton fabric obtained from single-bath treatment, the samples were subjected to whiteness index measurement using Macbeth spectrophotometer (Figure 3.4) to obtain the average whiteness value (front and back of fabrics). Measurement parameters were set up as follows: UV (excluded or included): 10^0 observer.

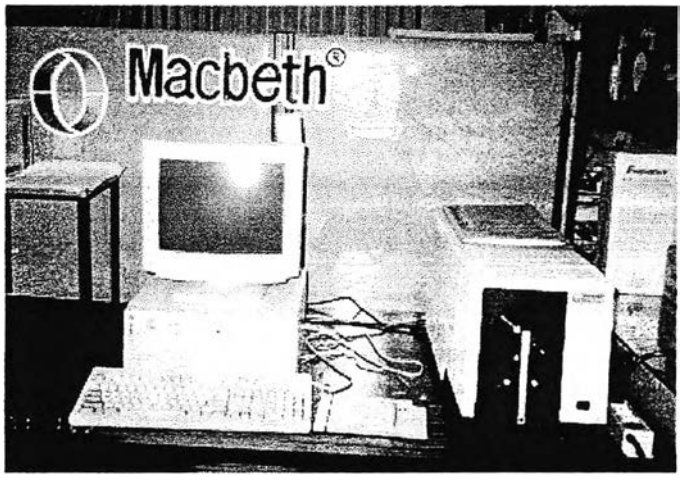

Figure 3.4 Macbeth spectrophotometer

3.4 Measurement of dye exhaustion

The total amount of dye taken by both modified and unmodified cotton fabrics was figured out by sampling the dye solution before and after the dyeing process. The absorbance of the equally diluted dye solutions was measured at the maximum wavelength of absorption of each dye using a UV/VIS spectrophotometer (Figure 3.5). The percentage of dye exhaustion (%E) was calculated with the following equation:

$$\%E = 100(1-A_1/A_0)$$

Where A_0 and A_1 are the absorbance of dye solution before and after dyeing process respectively.

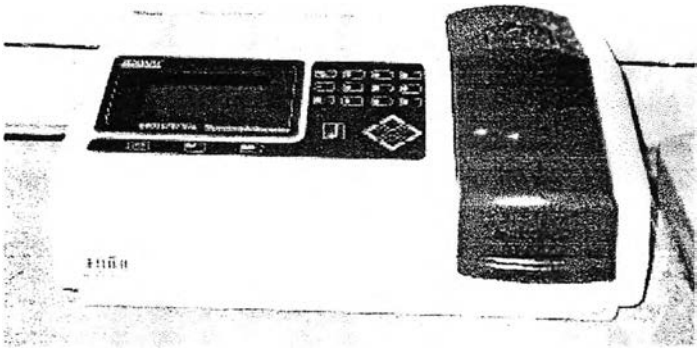

Figure 3.5 UV/Vis spectrophotometer

3.5 Measurement of dye fixation

The amount of dye fixed onto the fibers can be figured out by stripping of dyed fabrics in the solution of alkaline (Na_2CO_3) 5 g/l at the boil for 20 min. This result in the extraction of any unfixed dye from the dyed fabrics. By using an I.C.S. Micromath color measurement system (Macbeth 7000, Figure 3.4); the reflectance values (R) at the maximum wavelength of dyed fabrics before and after soaping were obtained.

$$K/S = (1-R)^2/2R$$

Where K = adsorption coefficient

S = scattering coefficient

R = reflectance

The development of the Kubelka-Monk equation in 1931 allows one to control and measure the opacity, reflectance, and color strength of dyes and dyed textiles.

It can be used in conjunction with CIELab systems or other color systems to match a given color standard.

By assuming K/S was proportional to dye concentration on the fibers, the extent of dye fixation could be calculated with the following equations:

$$\text{Degree of dye fixation (\%F)} = \frac{\text{K/S after soaping} \times 100}{\text{K/S before soaping}}$$

And

$$\text{Total dye fixation (\%T)} = \frac{\text{K/S after soaping} \times \text{(\%E)}}{\text{K/S before soaping}}$$

3.6 Total Nitrogen Content Determination

The total nitrogen content of treated fabrics was measured according to the rapid combustion method by an elemental analyzer (Perkin Elmer PE 2400 Series II) while mess index of bleached cotton fabric was measured using I.C.S Micrometer color measurement system (Macbeth 7000).

3.7 Image microscopic analysis (fiber cross sectional examination)

Although microscopic observations are extremely useful for determining numerous structural features and influences on fibers, careful sample preparation usually required is time-consuming and tedious. Thus, image analysis optically scanned with television camera was introduced as an alternative optical method to determine structural changes or staining in fibers.

Microscopic analysis of MAPTAC-treated dyed yarn was investigated using optical microscope (Figure 3.6). The fiber samples that sharply cut in perpendicular to the fiber axis were placed against transmitted light to be magnified by lenses and viewed on a screen to show the details of structural features, changes in fiber, and staining of dye.

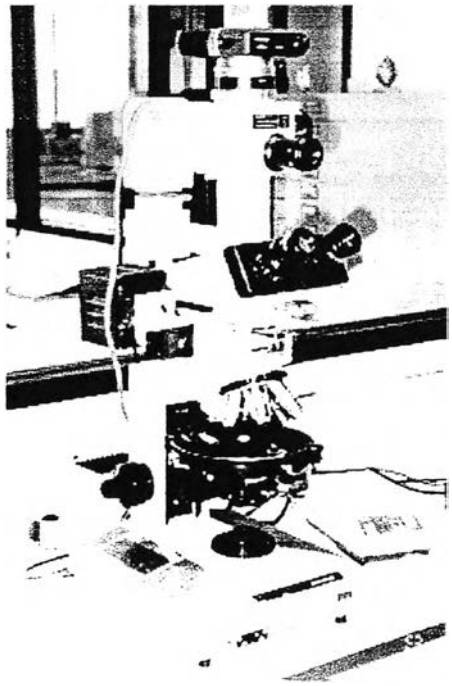

Figure 3.6 Optical microscope

3.8 Measurement of colorfastness

Fastness to light of modified and unmodified dyed cotton fabrics was conducted using Xenon Weather Meter (Figure 3.7), Model X75 (Suga Test Instruments Co., LTD, Japan) and following the ISO testing method. Blue wool fabrics were used as the standard of numerical ratings and were assigned to describe the colorfastness of the fabrics.

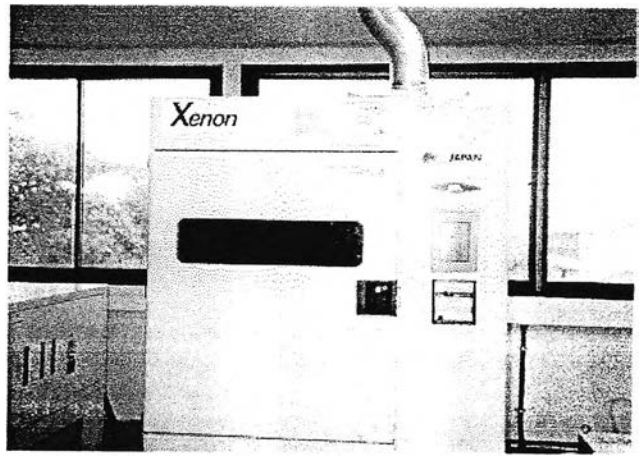

Figure 3.7 Xenon Weather Meter