CHAPTER III

Experimental Procedures

Scope

1. The 100% cotton fabrics which have already passed the pretreatment process will be crease resistant finished by pad-dry-cure method.

2. Citric acid was used as a cross-linking agent and sodium salts of phosphorus containing inorganic acids were used as the catalysts.

3. The fabrics were treated under various conditions by varying concentration of citric acid and catalyst as well as temperature and time of curing.

4. The physical properties of treated fabrics were determined and compared with commercial cross-linking agent by testing procedures as follow:

- Crease recovery angle
- CIE whiteness index
- Tensile strength

5. The properties of finished fabrics after repeated laundering have been determined at 1, 5, and 10 washing cycles by using launder-o-meter.

6. The esterification reaction between cellulose molecules and citric acid had been identified by FT-IR spectroscopic method.

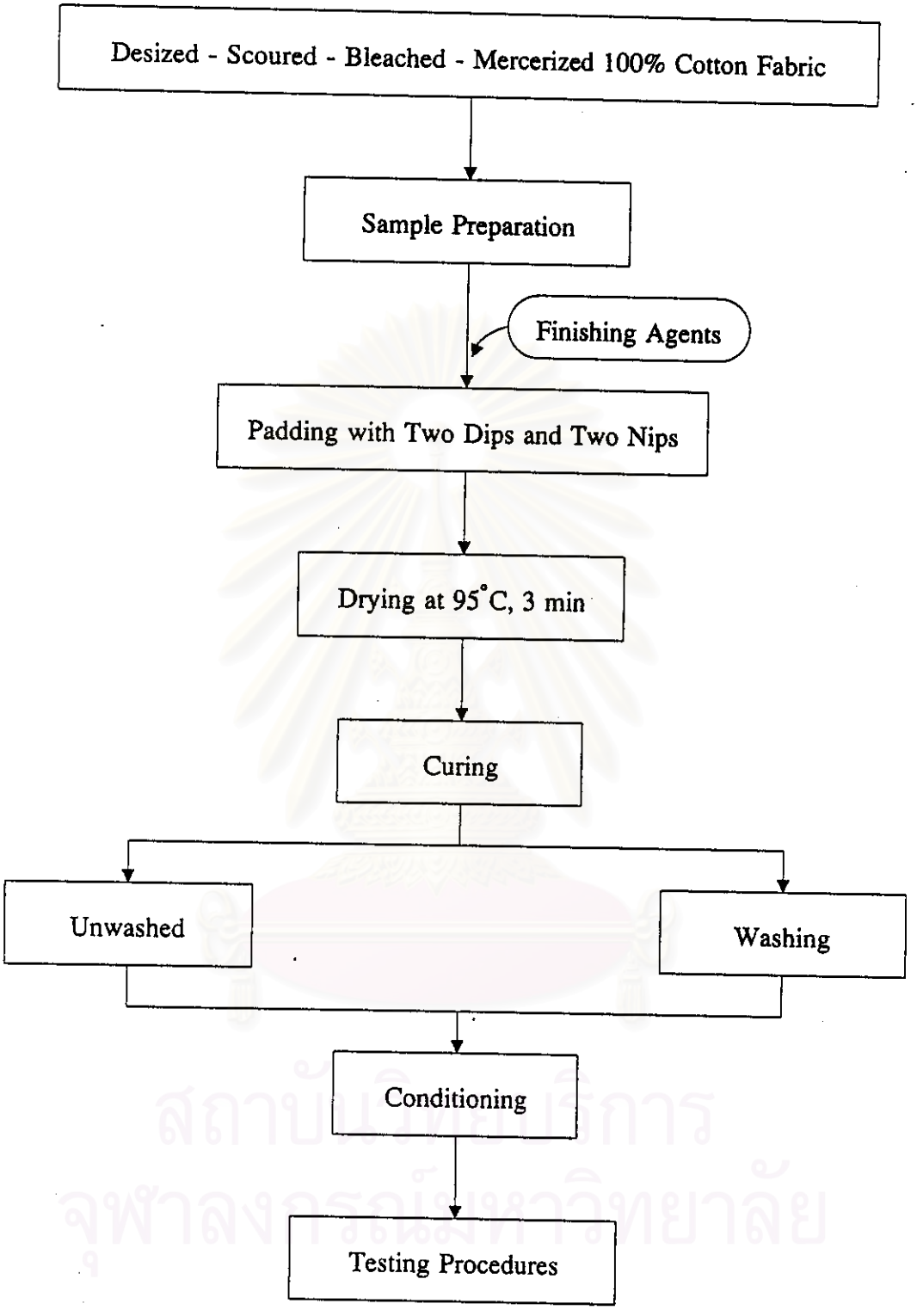

Figure 3.1 Flowchart of crease resistant finishing procedures.

Materials

The fabrics were desized, scoured, bleached and mercerized 100% cotton.

Chemicals

The chemicals used in these experiments were laboratory reagent grade.

1. Cross-Linking Agents

1.1 Citric Acid ($HOOC-CH_2-C(OH)(COOH)-CH_2-COOH$)
(MW = 210.14)

An organic acid, containing three carboxyl groups. In solid form, it is a water-soluble crystalline hydrate. Citric acid occurs in many fruits and vegetables. Its major uses are in food industry as an antioxidant activator, acidulant, soft-drink flavoring, color-fixing aid, etc. Moreover, it can act as a dispersing and sequestering agent and as a mordant in dyeing and textile printing (Hampel and Hawley, 1976).

1.2 Fixapret COC

For comparison with the commercial grade resin, Fixapret COC were used. It is the slightly yellowish transparent aqueous solution of a derivative of dimethylol-4,5-dihydroxy-ethylene urea (DMDHEU). It is recommended for low formaldehyde type resin finishing of white and coloured textiles composed of cellulosic fibres (Flick, 1990).

Active content : 40%

pH : 5.0-7.0

2. Catalysts

2.1 Tetra-Sodium Pyrophosphate Decahydrate. ($Na_4P_2O_7.10H_2O$)

(MW = 446.06)

Properties: Colorless, transparent crystals or white powder soluble in water, decomposes in alcohol.

Hazard: Toxic by inhalation (Richard, 1993).

2.2 Di-Sodium Hydrogenphosphate Dihydrate. ($Na_2HPO_4.2H_2O$)

(MW = 177.99)

Properties: Colorless, translucent crystals or white powder; saline taste. Soluble in water.

2.3 Sodium Dihydrogenphosphate Dihydrate. ($NaH_2PO_4.2H_2O$)

(MW = 156.01)

Properties: White, crystalline powder; slightly hygroscopic. Very soluble in water, has acid reaction.

2.4 Sodium Hypophosphite Monohydrate. ($NaH_2PO_2.H_2O$)

(MW = 106.06)

Properties: Colorless, crystalline plates or white granular powder; saline taste; soluble in water.

Hazard: Explosion risk when mixed with strong oxidizing agents, decomposes to phosphine on heating.

2.5 Condensol FB.

It is the tradename of special catalyst under BASF markets for effective cross-linking of cellulose fibers.

Equipments

1. Padding Mangle
2. Minidryer
3. Shirley Crease Recovery Tester
4. Reflectance Spectrophotometer
5. Instron Universal Tester
6. UV-VIS Spectrophotometer
7. Fourier Transform Infrared Spectrophotometer
8. Launder-o-meter
9. Pick Counter
10. Precision Balance
11. Glasswares

General Procedures for Crease Resistant Finishing Treatment.

1. Sample Preparation

The fabric used in the experiments has already passed the pretreatment processes, desize-scour-bleach-mercerize, but before entering the finishing process, starch on fabric must be checked by using iodine solution. Then the fabric treatments were carried out on a laboratory scale by cutting into sample (30x30 cm).

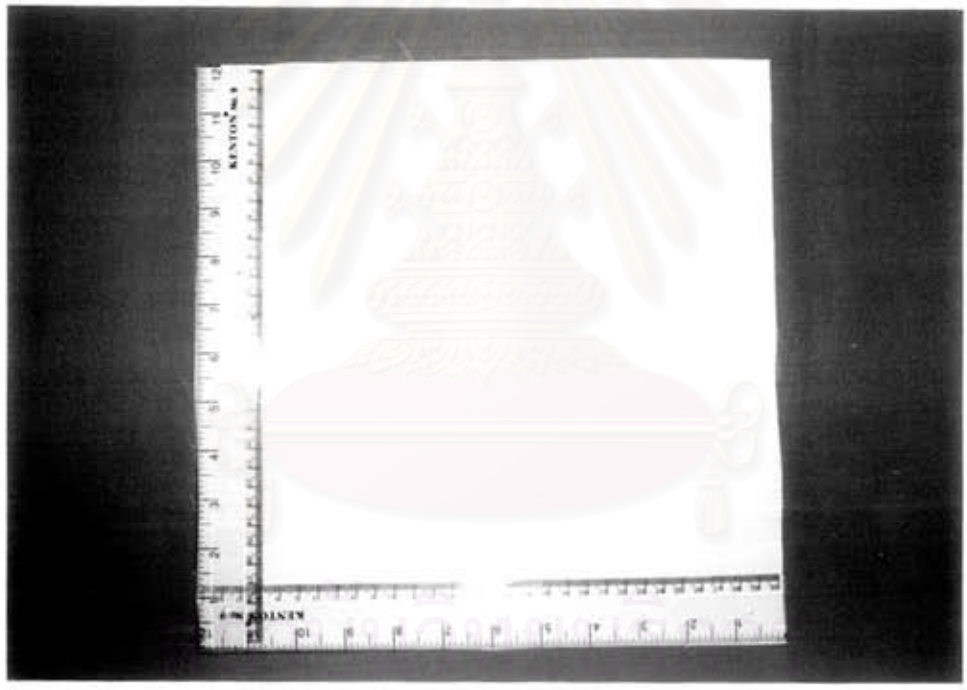

Figure 3.2 Sample of cotton fabric (30x30 cm).

2. Padding

The fabrics were treated with finishing agents by padding with two dips and two nips on a laboratory mangle to about 90% wet add-on.

Figure 3.3 Padding mangle.

3. Drying

The padded fabrics were dried at 95°C for 3 min by fixing on the tenter in the minidryer.

Figure 3.4 Minidryer.

4. Curing

Fabrics were cured at different cure conditions in the same minidryer.

5. Washing

The finished fabrics were washed by hand wash method at 60°C with a liquor-to-goods ratio of 25:1 and Lux flake detergent 2 g/l for 30 min, rinsed for 5 min and dried at 100°C for 2 min.

6. Conditioning

The finished fabrics were conditioned at 25±2°C, 65±2 %RH for at least 24 hours before testing.

General Procedures for Testing of Treated Fabrics.

1. Crease Recovery Angle.

1.1 Dry Crease Recovery Test

The method to determine dry and wet crease recovery of treated and untreated fabrics is the 'Shirley' crease recovery test (Booth, 1968).

Crease recovery angle of a specimen is the angle measured in degrees between the two edges of the test specimen after loading and recovery. Zero degrees crease recovery angle signifies no recovery from creasing and 180 degrees signified 100% recovery from creasing.

1.1.1 Apparatus.

1.1.1.1 The 'Shirley' crease recovery tester consists of a circular dial which carries the clamp for holding the specimen. Directly under the centre of the dial is a knife edge and an index line for measuring the recovery angle. The scale of the instrument is engraved on the dial (BS 3086 : 1972).

Figure 3.5 Shirley crease recovery tester.

1.1.1.2 A 2 kg weight with a convenient shape to provide uniform pressure over the area

1.1.1.3 A forcep with flat jaws.

1.1.1.4 A stop watch.

1.1.1.5 Glass plates (3 inches long by 1 inch wide).

1.1.2 Test Specimen.

Cut 20 specimens from the fabric that are free from wrinkle into 1 by 2 inch with a template, ten with their long dimension parallel to the warp and ten with their long dimension parallel to the filling.

1.1.3 Test Procedure.

1.1.3.1 Test the conditioned specimens in the standard testing atmosphere.

1.1.3.2 Bring the two edges of a specimen together and fold it exactly in half. Place it between two glass plates.

1.1.3.3 Put a 2 kg weight on a specimen.

1.1.3.4 After 1 min remove the weight.

1.1.3.5 Transfer a specimen to the clamp on the instrument by using a forcep and allow to recover from the crease for 1 min.

1.1.3.6 During Its recovery, the dial of the instrument is rotated to keep the free edge of the specimen in line with the knife edge.

1.1.3.7 At the end of the time period, the recovery angle in degrees is read on the engraved scale.

1.1.4 Report.

Warp and filling way recovery are reported separately in degree from the mean values of ten tests in each direction.

1.2 Wet Crease Recovery Test

The method of wet crease recovery test is in the same way as dry crease recovery test except putting the specimen into the distilled water for 1 min and quickly removing the excess water from the specimen before loading with 2 kg weight.

2. Tensile Strength. (AS L6 - 1970)

The tensile strength measurement was carried out by using the Instron Universal Tester (Model 5583) which is type E (constant-rate-of-extension). The test specimens were cut into strips, 60x300 mm and removing threads from

both edges until the width has been reduced to 50 mm. The number of specimen in warp and filling direction was 5 strips in each direction. The distance between the lower and the upper jaws was 200 mm. Each specimen was mounted centrally, gripped along the full width to prevent slipping. The use of soft packing between the specimen and the jaw faces can improve the occurrence of breaking close to the jaws. The machine was operated with a speed of 100 mm/min and the breaking load was read. The mean tensile strength value of warp and filling directions was separately determined in newton.

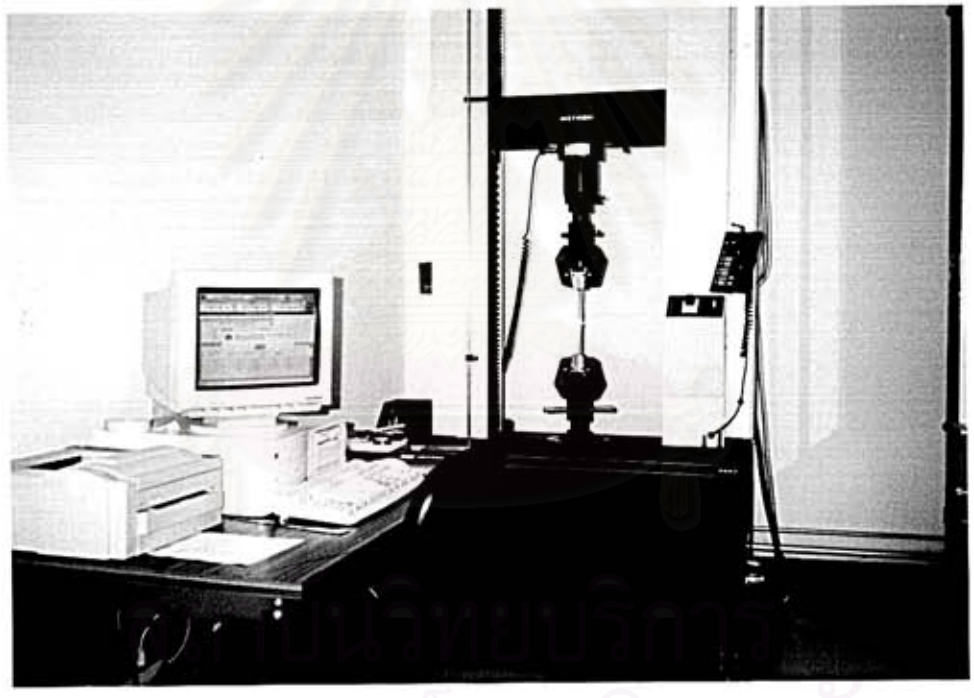

Figure 3.6 Instron universal tester, Model 5583.

3. Whiteness. (AATCC Test Method 110-1989)

Whiteness measurement is the method to indicate how white the textile appears to an average viewer. The CIE tristimulus values are measured using

a reflectance spectrophotometer, and the formulae for calculation of whiteness are those recommended by the CIE (Commission Internationale de l'Eclairage).

3.1 Apparatus.

Reflectance spectrophotometer with Xenon flash lamp capable of calculating CIE tristimulus values with D65/10°. Chromaticity co-ordinates are calculated from the tristimulus values automatically by the instrument.

Figure 3.7 Reflectance spectrophotometer.

3.2 Test Sample.

Keep the sample free of dirt. It is necessary to use the exact size of fabrics throughout the test and it must be large enough to cover the aperture of the reflectance measuring instrument.

3.3 Procedure.

3.3.1 After the instrument has been standardized according to the equipment's direction (Datacolor Spectraflash 500), fold the fabric sample until it is opaque (until an additional layers do not change the measurement). Be sure that there are no creases or marking in the measured area.

3.3.2 Make two measurements on each side of the fabric by rotated 90° between measurement on the same side.

3.4 Calculations and Interpretation.

For illuminant D65 and 10° observer, the whiteness index can be calculated by the following equation.

$$W = Y + 800 (x_0 - x) + 1700 (y_0 - y)$$

where :

W = whiteness index

Y = CIE tristimulus value of the sample

x,y = chromaticity co-ordinates of the sample

x_0, y_0 = chromaticity co-ordinates of perfect diffuser

($x_0 = 0.3138$, $y_0 = 0.3310$)

Comparison of whiteness indices should only be made when measuring samples at nearly the same time and on the same instrument. The higher the value of W, the greater the whiteness.

4. Determination of Cleavable Formaldehyde on Finished Fabrics (The Japanese Test Method Law 112-1973).

By this method not only formaldehyde actually present on the fabric is determined but also formaldehyde from partial hydrolysis during processing of sample by extraction (Petersen, 1985).

4.1 Reagents.

Ammonium acetate

Glacial acetic acid

Acetylacetone

4.2 Apparatus.

UV-VIS Spectrophotometer

Glass filters

Suction flasks, 250 ml

Erlenmeyer flasks with stopper, 250 ml

Pipets 100 ml, 5 ml, 2 ml, 1 ml

Beaker, 500 ml

Analytical balance

Volumetric flask, 1000 ml

Shaker

Test tubes with stoppers

4.3 Acetylacetone Reagent Solution.

150 g of ammonium acetate, 2 ml of acetylacetone and 3 ml of acetic acid are diluted up to the mark of a 1000-ml volumetric flask with distilled water.

4.4 Procedure.

A 250-ml Erlenmeyer flask is filled with 100 ml of distilled water via volumetric pipet. A precisely weighed fabric sample of about 1 g is placed in the Erlenmeyer flask, which is then closed with a stopper. The Erlenmeyerr flask is heated in shaker for 1 hr at 40°C. The solution is then filtered through a glass filter. The 5 ml filtrate is pipetted in a test tube and follow by 5 ml of acetylacetone reagent. The resulting solution is then heated to 40°C in shaker for 30 min. The absorbance is measured at 415 nm after cooling the solution to room temperature for 30 min.

4.5 Determination of a Calibration Curve.

A specific calibration curve has to be determined from a known formaldehyde solution. With each diluted solution, a color reaction with acetylacetone is carried out and the corresponding absorbance is recorded. The calibration curve is established by plotting the known concentrations versus the determined absorbance.

4.6 Preparation of the Standard Solution.

A 2-ml sample of formaldehyde solution (about 37%) is diluted to 1 liter with distilled water. The resulting solution is about 0.07% (740 ppm). The exact formaldehyde concentration is to be determined by titration, for instance by iodometric titration. After titration, 0.5, 1, 2, 3, 4, and 5 ml of the standard solution are pipetted into 500-ml volumetric flasks separately, which then

Figure 3.8 UV-VIS spectrophotometer.

are filled to volume by water. The formaldehyde content of these diluted solutions equals 0.7, 1.4, 2.8, 4.2, 5.6, and 7.0 ppm. The absorbance from these are determined by the acetylacetone method. In a test tube, 5 ml of the formaldehyde solutions and 5 ml acetylacetone reagent are added. The mixture is heated to 40°C for 30 min in a shaker and then cooled to room temperature. The absorbance is determined at 415 nm by using 5 ml of distilled water plus 5 ml of acetylacetone reagent solution as a blank.

4.7 Calculation.

Cleavable formaldehyde = C x 100/w ppm

where:

C = CH_2O concentration determined from calibration curve (ppm)

w = fabric weight (g)

5. Fourier Transform Infrared (FT-IR) Spectroscopy

As the classical molecular spectroscopic method, infrared (IR) spectroscopy is based on the interaction of infrared radiation with molecular dipole moments in the sample. Certain frequencies are absorbed and others are transmitted and a characteristic spectrum are, therefore, carried out for every substance (Willard, *et al.*, 1988).

The main components of a fourier transform infrared (FT-IR) spectrophotometer consist of the infrared light source, the interferometer, the sample chamber and the IR detector. When the radiation has passed through the interferometer and the sample, the signal "interferogram" will be generated at the detector. The infrared spectrum is calculated through fourier transformation of this data.

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Figure 3.9 . Fourier transform infrared spectrophotometer.

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 scans was 32. All the spectra were transferred to the computer for further analysis and plotting by Omnic software.