CHAPTER III

EXPERIMENTAL PROCEDURES

The experimental procedures have been explained under the following heads :

1. Materials used.
2. Determination of preliminary data of fabrics.
3. Preparation and application of finish.
4. Determination of wrinkle recovery.
5. Determination of tensile strength and elongation.
6. Analysis of formaldehyde.

1. Materials Used

Fabric used : White cotton cambric, commercially available was used in the study. The fabric was scoured with 2 gms/litre soap plus 2 gm/litre soda ash solution at 90°C keeping material to liquor ratio 1:30 for two hours. The fabric was then thoroughly washed and dried

Finishing substances used :

Finish used : Melamine formaldehyde*(Table 1).

Polyhydroxy alcohols used :

a) Diethylene glycol.

$HO(CH_2-CH_2O)_2H$

Mol. wt. 106.12

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* Superfinish MF, manufactured by Texchem, 132, Dr. Annie Besant Road, Bombay 400 018.

b) Polyethylene glycol-200

$HO\ (CH_2 - CH_2-O)_nH$

A.V.Mol. Wt. 190-220

c) Polyethylene glycol-400

$HO(CH_2-CH_2-O)_nH$

A.V. Mol. Wt. 380 - 420

Catalyst used :

Megnesiumchloride hexahydrate

$MgCl_26H_2O$.

2. Determination of Preliminary Data of Fabrics

(a) Determination of fabric count (9.0) :

The thread count of fabric was determined by counting the number of warp and weft threads per inch (converted to number of threads per centimeter) with the help of Alfred Suter Counter. Five readings were taken at different places at random in each of the warp and weft directions. Average of five readings was reported.

(b) Determination of weight per unit area of the fabric(9.0):

Five specimens, of size 5 cm. x 5 cm. were taken from different places of the cloth and were conditioned over saturated common salt solution in a desiccator. Each conditioned sample was weighed accurately on an analytical balance. Average of five readings was taken and was then calculated the weight per unit area in grams per square meter using the formula,

$$\text{Weight in gam/sq.m.} = \frac{W \times 100 \times 100}{5 \times 5}$$

Where 'W' is the average weight in gms of the specimens.

(c) Determination of thickness of fabrics (9a) :

Compressometer was used to determine the thickness of the fabrics. One specimen at a time was placed on the anvil without any folds and tension. The pressure foot was lowered upon the specimen by rotating the knob until upper dial read 5 (equal to 0.1 lb per square inch pressure) and the reading was reduced from the lower dial. The pressure was then increased until the upper dial read 40 (equal to 1 lb. per square inch pressure), and the lower dial reading was recorded again. The difference between the two readings gave the thickness of the fabric in inch (x0.001). The average of five readings was taken as the fabric thickness. This was converted to centimeters and reported to the nearest 0.001 cm.

3. Preparation and Application of Finish

The finish was applied on the fabric sample with the help of a laboratory padding mangle to get approximate 120 percent wet pick-up. The samples were dried at room temperature and then cured at 140°C for five and ten minutes and at 160°C for five minutes. A set of finished samples were given a standard wash with 2 g/l soap at 50°C for 30 minutes and used for analysis of total and liberated formaldehyde.

Recipe for various treatments given are :

(a) Treatment T_{MF} :

Melamine formaldehyde - 8 gm.
Megnesium chloride - 1 gm.
plus soft water to make total 100 ml.

(b) Treatment T_{1MF} :

Melamine formaldehyde - 8 gm.
Diethylene glycol - 2 gm.
Megnesium chloride - 1 gm.
plus soft water to make total 100 ml.

(c) Treatment T_{2MF} :

Melamine formaldehyde - 8 gm.
Polyethylene glycol-200 - 2 gm.
Megnesium chloride - 1 gm.
plus soft water to make total 100 ml.

(d) Treatment T_{3MF} :

Melamine formaldehyde - 8 gm.
Polyethylene glycol -400 - 2 gm.
Megnesium chloride - 1 gm.
plus soft water to make total 100 ml.

4. Determination of Wrinkle Recovery (4).

Wrinkle recovery was determined using AATCC test method 66 - 1968, wrinkle recovery angle method. Samples measuring

4.0 cm x 1.5 cm. were cut from untreated and treated samples, three for the face to face and three for back to back tests in warp and weft directions. The samples were conditioned over saturated common salt in a desiccator for twelve hours before testing. The test specimen was placed between specimen holder. The exposed end of the specimen was folded back to the guide line on the shorter, thin metal leaf and held there firmly with the left thumb nail, the specimen and holder was inserted between the jaws of the plastic press in such a manner so that the jaw having the small raised platform was outside and parallel to the longer metal strip of the holder.

The press holder combination was inverted on a table with small platform upward, and a load of 500 gm. was applied to the platform, load was removed exactly after 5 minutes and then the press holder combination was inserted with the exposed end of the specimen holder in the mount on the face of the tester. The press holder was removed from the jaws, after which the specimen holder was properly aligned on the mounting shelf. The crease was lined with the mark at the centre of the tester disc, and the dangling specimen leg was lined up immediately with the vertical guide line. In order to eliminate gravitational effects, the dangling specimen leg was kept aligned with the vertical guide line during the five minute recovery period, After five minutes of recovery a final adjustment of the dangling let to the vertical guideline was made, and the crease recovery angle was read from the protractor,

scale. Average of individual warp and weft readings were reported seperately. Total crease recovery was calculated by the addition of average warp and weft crease recovery.

5. Determination of Strength and Elongation (9,6) :

The tensile strength and elongation of the untreated and and treated samples were determined on the Scott Tester. Samples of 15 cm. x 3 cm. were cut at random in the warp direction. They were ravelled from both sides to measure 2.5 cm. The samples were conditioned over saturated common salt solution. Gauge length was kept at 7.5 cm. The breaking length and elongation were noted from the graph. An average of six readings was taken. The percentage elongation at breaking point as well as at intermediate loads were calculated from the formula :

$$\text{Percentage elongation} = \frac{Y \times 100}{X}$$

Y = Elongation obtained from the graph.

X = Length of the specimen (7.5 cm.)

6. Analysis of Formaldehyde :

(a) Analysis of total formaldehyde (33) :

Treated fabric sample weighing 100 mg. was taken in a 100 ml. capacity conical flask at 25 ml. of 12 N-sulphuric acid was added, stoppered at kept overnight for hydrolysis. One ml. of aliquot was taken in a test tube with 50 ml. mark, to this 0.5 ml. of chromotropic acid (10% conc.) was added

followed by the addition of 5 ml. concentrated sulphuric acid. The test tube was kept in a boiling water bath for 30 minutes and then cooled. The solution was diluted to 50 ml. and the solution was cooled to room temperature. The optical density of colour developed was measured using colorimeter with filter at 570 m/r. Any dilution needed to get reading in desired range were made before determining optical density. An average of two sets of readings was taken. The mg. of formaldehyde in one ml. was read from the calibration curve of optical density vs mg. of formaldehyde and final concentration of formaldehyde was reported in parts per million (PPM).

$$PPM = \% \text{ Formaldehyde} \times 10^4 = mg/kg.$$

(b) Analysis of free formaldehyde (1) :

Of the finished fabric one gm. of sample was accurately weighed and taken into 500 ml. capacity jar, 100 ml. of distilled water was added and the jar was tightly closed and kept at 40°C for 60 minutes. The jar was shaken repeatedly by hand. The content of jar was centrifuged and one ml. of aliquot was used for analysis of formaldehyde as described above. An average of two sets of readings was taken.

(c) Analysis of liberated formaldehyde (5) :

Liberated formaldehyde was analysed using A.A.T.C.C. Test Method - 112 - 1968. This sealed jar method provides an analytical means for estimation of the amount of formaldehyde

released under conditions similar to those of actual storage. One gram of finished sample was suspended inside a glass jar in which 50 ml. of distilled water was at the bottom. The glass jar was sealed and placed in the oven at 50°C for 20 hours. After the duration the sample was taken out of the jar. The jar was resealed and shaken to mix the condensation formed on sides of the jar. One ml. of aliquot portion from the jar was used for formaldehyde analysis using chromotropic acid method as described above. Average of two sets of readings was taken.