CHAPTER II

MATERIALS AND METHODS
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MATERIALS

2.1 Fabric

White cotton poplin having the construction 36/30 counts and 112/72 ends/oicks per inch was used in the present investigations.

2.2 Soils

The following materials were used as soils:

2.2.1 Particulate Soils

(a) Ferric oxide (red) powder of A.R. grade with particle size ranging from 0.2 to 1.2 micron,

(b) Carbon black of four particle ranges 2-10, 40-50, 50-75 and 75-150 microns respectively, and

(c) Air-borne natural particulate soil (NPS) collected during dry weather from the laboratory premises.

2.2.2 Oily Soils

(a) Lubricating oil of the grade (SAE 40) having viscosity 220 cps and density 0.898 g/cm$^3$ at 27°C, and

(b) Used lubricating oil from the same grade having viscosity 275 cps and density
0.909 g/cm³ at 27°C. The lubricating oil was obtained from Indian Oil Corporation Limited, Bombay.

2.2.3 Synthetic Sebum

Synthetic sebum was prepared by dissolving glyceryl tristearate (30 parts), stearic acid (30 parts), octadecyl alcohol (20 parts) and octadecane (20 parts) in required quantity of carbon tetrachloride to yield 5, 10, 20 and 50% w/w concentrations of the composite mixture.

2.3 Surfactant

Sodium dodecyl sulfate (SDS) having molecular formula C_{12}H_{25}SO₄Na and molecular weight 288.4, in the form of pure white flakes, made by Koch-Light Pvt. Ltd., U.K., was used as surfactant.

2.4 Additives in Washing Bath

The following compounds were used as additives along with washing solution as and when required:

2.4.1 Sodium tripolyphosphate (STPP)
2.4.2 Carboxymethylcellulose (CMC)

2.5 Crosslinking Agent

N,N'-Dimethylol 4,5-dihydroxy ethylene urea (DMDOH) obtained from Shri Ambuja Chemicals Co., Ahmedabad, was used as the crosslinking agent for the poplin. The analytical
data of the DMBHEU were as below:

Solid content = 41.0 %
Nitrogen content = 6.2 %
Free formaldehyde content = 1.2 %
Bound formaldehyde content = 13.4 %

2.6 Additives in the Crosslinking Bath

The following additives were used in the crosslinking bath optionally:

2.6.1 Polyvinyl acetate dispersion (PVAD)
2.6.2 Silicon emulsion (SE)
2.6.3 Polyethylene emulsion (PEE)
2.6.4 SLN Binder (SLNB)

The first three products were obtained from HICO Products Pvt. Ltd., Bombay. The fourth one was obtained from ATIRA, Ahmedabad.

2.7 Miscellaneous Chemical

2.7.1 Bakcar Print Blue VR

An oil soluble dye obtained from Du Pont Co., U.S.A., was used to dye colourless oily soils at concentrations of 0.2% w/w. The active ingredient of the dye was Milling Blue BL Base, obtained from Du Pont, U.S.A.
2.8 Apparatus

2.8.1 Soiling Machines

(a) Accelerator

The instrument specified as Type AB 7 of Atlas Electric Devices Co., Chicago, U.S.A., with a 4½" rotor (elongated S-shape, offset) without the abrasive Liner was used for soiling fabrics with particulate soils.

(b) FIRA Soiling Tester

The instrument obtained from Shirley Developments Ltd., Manchester was used for soiling with particulate soil. The instrument was equipped with eight metal cylinders (10.0 cm diameter and 40.0 cm length, each) and two protective guards. A set of 350 woollen felt cubes (1 cm³ size, 1 g. weight, each) was also provided along with the instrument.

2.8.2 Washing Machine

Tergotometer, Model TM-4, made in Japan, was used as a washing machine.

2.8.3 Spectrophotometers

(a) Beckman Spectrophotometer

Model DU of Beckman Instruments Inc., Fullerton, California, was used with reflectance measuring attachment.
(b) Reflection Meter

Model CG-166, Ser. 1480 of Canadian Research Institute, Canada with three tristimulus filters, namely, green, blue and amber was used for reflectance measurements.

(c) Spectronic 20

The model of Bausch and Lomb, U.S.A., was used for colourimetric analysis.

2.8.4 Scanning Electron Microscope

Cambridge Stereoscan, Model S4-10, U.K., was used for microscopic observations of soils and fabrics.

2.8.5 Miscellaneous Apparatus

(a) Micrometer Syringe

A gas-tight syringe (0.5 ml) attached with a micrometer device specified as 'Agla' brand of Burroughs Wellcome and Co., London, U.K., was used to obtain minute quantity of oily soils with accuracy of 0.01 ml.

(b) Wooden embroidery hoops with 12.0 cm internal diameter were used for mounting fabrics for soiling with oily soils by wicking method.

(c) Stroboflash, Type 1200D of Dewe Instruments Ltd., London, was used for calibration of the Accelerotor.
METHODS

2.1 Fabric Treatments

The crosslinking treatments were given to one meter long fabrics. The fabric samples were padded with resin solution (material to liquor ratio 1:10) containing specific crosslinking agents, required catalyst and other additives (optionally) to 75% wet pick up. The samples were dried at 110°C for 1 minute and cured at 150°C for 4 minutes in a Monfort single compartment stenter. The cured samples were washed with 2 g/1 sodium carbonate solution at 50°C for 20-30 minutes. Then they were rinsed successively with tap water, and distilled water, dried in air and conditioned at 65% R.H.

2.2 Soiling Methods

2.2.1 Particulate Soils

(a) The Accelerotor Method

The speed of Accelerotor was calibrated against Stroboflash.

Two 10 cm x 10 cm samples of the fabric were cut and weighed. The amount of soil was calculated from the weight of samples (1-20% owf) or kept constant (5% owf). The soil was weighed accurately.

The rotor of Accelerotor was turned to the
horizontal position and the two samples were placed as one on each end of the rotor. The door of the chamber was closed and the soil was put inside through the upper hole of the chamber. The time of soiling was monitored by a stop-watch (usually 1 minute). The selected speed of 1600 rpm was maintained as closely as possible. After soiling, the soiled samples and residual soil were taken out from the chamber.

The excess or 'loose' soil was removed by shaking the soiled samples once, followed by a simulated vacuum cleaning. In this procedure, a soiled sample was clamped with paper-clips on a wire screen. The funnel as specified was attached to vacuum pump through suitable traps. The soiled sample was cleaned by sliding the funnel with parallel strokes on the fabric surface for 30 seconds in one direction and 30 seconds in crosswise direction. Both the sides of the two soiled samples were cleaned in the same manner.

(b) The FIRA Tumbler Method

The soiling machine consists of eight metal cylinders mounted radially. Each cylinder possesses removable lids at both the ends with sample-retaining plastic rings. The cylinders were rotated at 24 ± 2 rev/min by an electric motor.
The direction of rotation is changed every 10 minutes by means of a timed electrical reversing switch.

In this method, transfer of soil takes place through felt cubes.

The amount of soil was calculated from the weight of the fabric samples (5-30% owf). The soil was applied to felt cubes by tumbling the soil and the cubes for 12 cycles of 10 minutes duration each. The excess soil was removed from the cylinder. Two circular fabric samples (9.0 cm diameter each) were mounted inside the lids at the end of the cylinder. The soiled cubes were placed into the cylinder and tumbled for 9 to 18 cycles. After the soiling was over, the samples were removed from the cylinder. Unless stated otherwise, the amount of soil taken for soiling 40 cubes was 10% owf, the cubes soiled for 12 cycles and the fabric samples for 14 cycles.

The cubes were charged with soil after each soiling test. Eventually, when the cubes become worn they should be discarded. During the rotation, the cylinders were always covered with two protective guards.

The excess soil on the samples, if any, was removed by shaking the samples once.
2.2.2 Oily Soils

(a) The AATCC Test Method 130/1970

Three fabric samples (15 cm x 15 cm) for each determination were conditioned at 65% RH for 12 hours. Then they were placed on a textile blotting paper on a glass plate. In the centre of the sample, 0.2 ml of oily soil was placed. A glassine paper (7.5 cm x 7.5 cm) was placed over the oil puddle followed by an iron cylinder of 5 lbs weight for 60 seconds. Then the weight was removed and the glassine paper was discarded. The samples were washed within 15 to 60 minutes after soiling.

(b) The Wicking Method

Two fabric samples (15 x 15-cm) for each determination were cut and mounted separately on embroidery hoops. Then they were placed in a desiccator and were equilibrated to dry condition for 12 hours. A known quantity of oily soil (0.10-0.80 ± 0.005 ml) was placed on the centre of the mounted samples by means of a micrometer syringe. The oil was allowed to wick for a period of 15 hours under the same condition.

2.2.3 Aqueous Soiling

A known amount of particulate soil was added to water (2-10x10^{-2} %). The mixture was taken into the
beaker of Tergotometer and was agitated vigorously at
120 rpm for 15 minutes in order to obtain a homogeneous
dispersion of the particulate soil. Then an unsoiled
sample in wet condition was inserted and was agitated slowly
at 30 rpm for 30 minutes. After the soiling, the sample
was taken out of the beaker. It was then rinsed in running
tap water for 5 minutes followed by rinsing with distilled
water and dried in an electric oven at 60°C for 20-25 minutes.

2.3 Washing Method

Each wash-beaker of Tergotometer was usually filled
with 250 ml of surfactant solution with desired concentra-
tion and the entire wash-system was maintained at a constant
temperature in the range of 35-85°C ± 0.5°C.

The fabric sample to be washed was inserted in each
wash-beaker and then the wash liquor was agitated at 20-80
rpm for 10-60 minutes. After the washing was over, samples
were rinsed separately in running tap water with constant
flow for 5 minutes followed by rinsing with distilled water.
Then they were dried in an electric oven at 60°C for 20-25
minutes.

2.4 Estimation of Soil on Fabric

2.4.1 Reflectance Measurements

The reflectance of soiled and unsoiled fabric
samples before and after washing was measured with Beckman
DU spectrophotometer and Reflection Meter. The latter one
was used with three tristimulus filters, namely, green,
blue and amber. Both the instruments were calibrated against their respective white standards.

In case of Beckman DU, four readings were made on each side of each sample but an overall one reading on each side of each sample was obtained in case of Reflection Meter. In all cases, an average value of reflectance was considered.

The reflectance data were converted to Kubelka-Munk (Ka/Ks) ratios:

$$\frac{Ka}{Ks} = \frac{(1-R)^2}{2R},$$

where Ka is the absorption coefficient, Ks is the scattering coefficient and R the reflectance of the fabric (expressed as a decimal fraction).

The amount of soil on fabric was expressed as soiling value as follows:

- the soiling value of the soiled fabric before washing,
  $$V = (\frac{Ka}{Ks})_v - (\frac{Ka}{Ks})_i,$$
  (particulate soils);
- the soiling value of the soiled fabric after washing,
  $$W = (\frac{Ka}{Ks})_w - (\frac{Ka}{Ks})_u,$$
  where, \((\frac{Ka}{Ks})_v, (\frac{Ka}{Ks})_i, (\frac{Ka}{Ks})_s, (\frac{Ka}{Ks})_w\) and \((\frac{Ka}{Ks})_u\) are the Kubelka-Munk ratios of vacuum-cleaned, initial, soiled with oily soil, washed and unsoiled but washed fabric samples respectively. Further, the percent soil retained was calculated as 100 W/V or 100 W/S.
The tristimulus values were calculated by the following relationships:

\[
X = 0.783 \, R_x + 0.197 \, R_z \\
Y = R_y \\
Z = 1.181 \, R_z
\]

where \( R_x, R_y \) and \( R_z \) are the reflectance values obtained with the amber, green and blue filters, respectively. The degree of soiling was calculated as the difference between the values before and after soiling.

### 2.4.2 Chemical Analysis

The ferric oxide content of the fabrics was determined by ashing the fabric with sulfuric acid and analyzing for iron spectrophotometrically by the 1,10-phenanthroline method.

### 2.4.3 Gravimetric Analysis

The portion of fabric covered by the oil spot was cut out carefully and weighed. Then the oil was extracted from fabric with chloroform in a soxhlet extraction unit for 4 hours. The fabric sample was dried and weighed again. The oil was also obtained by removing chloroform and was weighed. The oil density per unit mass of the fabric was calculated from the weight difference.
2.4.4 **Statistical Analysis**

The correlation coefficient (r) and the standard deviation (sigma) was calculated by standard formulas. The reliability was computed at 95% confidence level.

2.5 **Microscopy**

The scanning electron microscope (SEM) was employed for observation of soils and fabrics. The size of particulate soils was also determined by SEM. All the samples prior to observation were coated with gold in a vacuum coating unit. The magnification is indicated with respective micrographs.

2.6 **Fabric Testing**

2.6.1 **Physical Testing**

(a) **Dry Crease Recovery**

Dry crease recovery angles (warp + weft) of fabrics were measured according to Monsanto method. Each result given is the average of 10 samples (5 samples with warp threads creased and 5 samples with weft threads creased).

(b) **Moisture Regain**

Moisture regain values were determined by drying fabric samples at 110°C for 4 hours and then equilibrating them for 24 hours at 65% RH.
The percent moisture regain was calculated from the change in weight before and after equilibration.

(c) Vertical Wicking Test

The fabric sample to be tested was rinsed in deionized water at 50°C for 3 hours. Then it was dried at room temperature. From the sample three strips of 10 cm x 1.25 cm size each were cut warp-wise and conditioned at 65% RH and 27°C. Then a strip was weighed accurately and suspended vertically in an air-thermostat maintained at 65% RH with a glass panel in the front. A trough containing distilled water was kept on a jack below the suspended strip. The height of the jack was adjusted so as to bring the level of the water just touching the lower end of the strip. The rise of water on the strip was measured as a function of time with the help of a cathetometer. Observations were made till the water-level ascended 5.0 cm above the lower end of the strip. The time required to ascend this height was recorded. The strip was then weighed with minimum exposure to reduce any error caused by the evaporation of water. From the difference in the initial and the final weight of the strip, the amount of water (% owf) taken up by the strip was calculated. The same procedure was repeated for two more strips of the same fabric sample and an average value was considered.
2.6.2 Chemical Testing

(a) Nitrogen Content

Nitrogen content of treated fabric was determined by the Kjeldahl micro method.
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