CHAPTER IV

EXPERIMENTAL PROCEDURE

The procedure has been divided into the following sub-sections:

4.1 Determination of preliminary data of the fabrics.

4.2 Determination of related properties of soaps, detergents and their combinations (surface tension, wettability, wetting time, emulsification and foaming power).

4.3 Preparation of soil and preparation of soiled samples.

4.4 Laundering procedure for soiled samples.

4.5 Determination of reflectance of soiled and washed samples.

4.6 Analysis of results to get the optimum combination composition.

4.7 Fabrication of a small washing machine to study the applications of cleaning solutions in washing.

4.1 Determination of preliminary data of the fabrics

Three commercially available fabrics were used. These were cotton (poplin), polyester/cotton (67/33 blend) and polyester fabrics.

The preliminary data of these fabrics on weight per unit area, thread count and thickness were determined as per standard procedures given below.

4.1.1 Determination of weight of fabric per unit area

Five specimens, of 5 cms x 5 cms were cut at random from each fabric. The specimens were conditioned over saturated common salt solution (65% RH) at room temperature, in a glass cabinet for 24 hrs. Each sample was weighed on an analytical balance. An average of five readings was obtained. Weight per unit area in oz. per sq.yd. was calculated using the following formula:

$$\text{Weight per unit area (oz./sq.yd.)} = \frac{W(\text{gm.}) \times 36(\text{inch}) \times 36(\text{inch})}{2(\text{inch}) \times 2(\text{inch})} \times \frac{1}{28.4}$$

It was reported in gms per square meter, calculated from the formula:

$$\text{Weight (gm./sq.m.)} = \frac{W(\text{gm.}) \times 100(\text{cm}) \times 100(\text{cm})}{5(\text{cm}) \times 5(\text{cm})}$$

4.1.2 Determination of thread count of fabrics

The number of threads per inch in the warp and the weft direction were counted, using Alfred Suter Counter. An average of five readings was taken. The data was also reported in the metric system (number of threads per cm).

4.1.3 Determination of compressible thickness of the fabrics

The Compressometer was used to determine the compressible thickness of the fabrics. A specimen was placed on the flat surface, below the pressure foot of the instrument. The pressure

foot was lowered upon the specimen by rotating the knob slowly, until the upper dial read 5 (equal to 0.1 lb. pressure per square inch) and the reading was recorded from the lower dial. The pressure was increased until the upper dial read 40 (equal to 1.0 lb. pressure per square inch) and the lower dial reading was recorded again. The difference between the two lower dial readings gave the compressible thickness of the fabric (x 0.001 inch). The average of five readings was taken. This was also converted into metric system and reported to the nearest .001 cm.

4.2 Determination of related properties of soaps, detergents and their combinations

(surface tension, wettability, wetting time, emulsification and foaming power)

Two soaps and two anionic synthetic detergent and one nonionic detergents were used for this study. The two soaps were commercial 501 bar soap (purified) and sodium oleate, the two anionic synthetic detergents were Teepol (purified) and sodium lauryl sulphate and the nonionic detergent was Lissapol N. These were used alone and in combinations. The concentrations studied were 1.0, 2.0, 3.0, 4.0 and 5.0 g/l for soaps and synthetic detergents (individual). Their combinations were varied in percentage ratio of 75:25, 50:50 and 25:75, the concentrations being 1.25, 2.5 and 5 g/l.

The combinations studied were :

Combination I	-	501 bar soap and Teepol
Combination II	-	Sodium oleate and Teepol
Combination III	-	501 bar soap and sodium lauryl sulphate
Combination IV	-	501 bar soap and Lissapol N.

Sodium oleate, a costly soap, was used in only one combination viz. II and served as a comparison.

All the solutions were prepared in soft water, and their pH was noted with the use of pH paper.

Purification (31)

The commercial 501 bar soap was purified to remove the builders. Teepol was also purified to remove the neutralized salts. The others did not need purification and hence were used as such.

Purification of soap

10 gms of grated soap was pasted with 10 ml of water. Then 90 ml of 95% ethyl alcohol (rectified spirit) was slowly poured with continuous stirring. The solution was allowed to settle the builders and impurities. The solution was then filtered to remove builders and precipitated impurities. The pure soap was kept in a weighed dish so that the alcohol would evaporate. This was again weighed. The amount of pure soap and the amount of builders were calculated.

Purification of Teepol

10 ml of Teepol was dissolved in 20 ml of 95% ethyl alcohol (rectified spirit) and was allowed to settle. It was then filtered to remove the salts. The solution was kept in a dish to evaporate the alcohol.

4.2.1 Determination of surface tension (3)

Surface tension is a primary property of any surface active agent and plays an important part in detergency. Surface tension for the present study was determined by the drop method. Solution to be tested was taken into a burette. The stopper of the burette was opened to release the drops slowly and the number of drops in 5 ml was counted. The number of drops are inversely proportional to the surface tension. The relative surface tension was calculated using the formula given below:

$$T_1 = \frac{N_2}{N_1}$$ where,

T_1 - surface tension of test solution

N_1 - number of drops in test solution

N_2 - number of drops in water.

The surface tension was then converted into dynes/cm by multiplying by 72.8 the surface tension of water being 72.8 dynes/cm.

4.2.2 Determination of percentage wettability (3)

Percentage wettability is the amount of solution that penetrates the sample under conditions of wetting. Therefore if the fabric is kept constant then one can determine which cleansing agent is absorbed better than another one, a good absorbancy helps cleaning.

The wettability was determined by the centrifuge method. Samples of 5 cm x 5 cm were cut of scoured grey fabric, and weighed individually on an analytical balance. The samples were wetted in the test solution for 5 mins. Two samples at a time were then centrifuged on a hand centrifuge for one minute at approximately 1400 rpm. They were weighed again. The increase in weight converted to percentage or original weight gave % wettability. An average of three readings was recorded. This is also called the Herbig number method and also adopted by the Indian Standards Institution. Herbig method is however automatic centrifuging after wetting.

4.2.3 Determination of wetting time (7)

Wetting time was determined by the drop method as a drop of the test solution to wet a sample. The fabric (scoured grey cotton) was fixed in an embroidery ring. The test solution was taken in a burette, and the tip was adjusted to one inch above the fabric. A drop was allowed to fall on the surface of the

fabric and the stop watch was immediately started. The stop watch was stopped when the drop lost its specular reflective power. A source of light was placed behind the fabric as an aid to see the reflectance. An average of three readings was taken and recorded as wetting time in seconds.

4.2.4 Determination of foaming power (3)

The test for comparing the foaming power of surface active agents involves shaking solutions by hand in a glass stoppered measuring cylinder. 30 ml of test solution was taken in a 100 ml cylinder. The cylinder was stoppered and carefully shaken by hand up and down, 30 times using as nearly as possible the same arm action it was then placed vertical on the table. The height of the foam was read after 30 secs, 2 mins, 5 mins and 10 mins to note the amount of foam and its stability. The foaming power was noted as foam height, in cms. An average of three readings was taken.

4.2.5 Determination of emulsifying power (3)

The procedure for determining the emulsifying power of cleaning agent was as follows:
Five ml of test solution was taken and one ml of soil mixture containing oils, colouring matter and kerosene was added. A graph paper strip with cm markings was pasted on the test tube which helped in taking the readings. The test tube was shaken

10 times up and down and was allowed to stand for 10 mins. The emulsified layer was measured in cms at 2 mins, 5 mins and 10 mins.

4.3 Preparation of soil and preparation of soiled samples

Preparation of fabrics for soiling

The cotton fabric (poplin) was scoured at 80-85°C for 45 mins with solution containing 5 g/l of 501 bar soap and 5 g/l of soda ash. The polyester/cotton fabric was scoured at 60-80°C for 30 mins with solution containing 2.5 g/l of 501 bar soap and 2.5 g/l of soda ash. Polyester fabric was scoured at 45-60°C for 30 mins with solution containing 2.5 g/l of soap and 2.5 g/l soda ash. The material liquor ratio was maintained at 1:30 for all the fabrics. After scouring, fabrics were rinsed thoroughly in water and air dried.

Fabrics were soiled by two methods : (a) solvent soil method and (b) emulsion soil method.

Solvent soil

The solvent soil was prepared as follows:

Carbon black (Jai Kajal)	..	2 gm
Coconut oil ..	..	5 ml
Stearic acid ..	..	2 gm
Oleic acid ..	..	10 ml
Carbon tetrachloride	..	85 ml
		100 ml

Carbon black was mixed thoroughly with coconut oil; stearic acid and oleic acid were mixed. Then the two parts were mixed together. Carbon tetrachloride was then added and stirred well.

Emulsion soil

The emulsion soil was prepared as follows:
The soil mixture was prepared and added to soap solution along with kerosene and emulsified as given below:

Soap solution (5 g/l)	..	50 ml
Soil mixture	..	30 ml
Kerosene	..	20 ml
Total	..	100 ml

Soil mixture:

Coconut oil	..	10 ml
Oleic acid	..	20 ml
Stearic acid	..	2 gm
Carbon black (Jai Kajal)	..	2 gm
		30 ml

Coconut oil and carbon black was mixed thoroughly and oleic acid and stearic acid were mixed together, the two were mixed together. The soil mixture was added to kerosene. This was then added slowly to the soap solution with continuous stirring. The stirring was continued for getting an even suspension.

Purpose of the ingredients

1 Carbon black is used as a grey coloured pigment to indicate greyness. (Jai Kajal manufactured by Western India Chemical Co., Bombay)

2 Coconut oil is used because it does not leave any ageing effect and it helps in dispersing pigments in the solvent.

3 Oleic acid is used as it helps in even dispersion of soil particles.

4 Stearic acid is similar to oleic acid and simulates wax.

5 Carbon tetrachloride is used as a solvent.

6 Kerosene is used because it gives dispersion of soil in the soap solution and allows it to go on to the fabric for emulsion soiling.

7 Soap solution is used for getting an emulsion.

Procedure of soiling - Soiling was done by passing the scoured sample through the soil solution, taken in an enamel tray. Glass rods were kept on top of the fabric so as to move away the excess soil solution and so get even soiling. This simulates padding with little pressure. Samples were hung in air for drying.

4.4 Laundering of soiled samples (7)

The soiled samples were cut to 2.5 inch x 5 inch and were stitched to white (scoured) fabric of the same size. (This was done to see if during washing there was any staining or redeposition of the soil on the white sample.)

The different detergent solutions as required, were prepared in soft water.

To assess the cleaning efficiency, the washing tests were carried out using the Launder-Ometer for washing. Launder-Ometer is an instrument manufactured by Atlas Electric Devices Co., Chicago. It has been widely adopted because it is well designed, to control the mechanical and physical factors that effect the washing process - namely time, temperature and degree of agitation.

Bottles with the test solutions (300 ml), samples (3 samples of 5 inch x 5 inch) and 1/4 inch diameter steel balls (40) were rotated for 15 minutes at room temperature. The steel balls were to provide agitation as jars turned. The material liquor ratio was kept at 1:30. The samples so washed were removed from the jars after 15 mins and were rinsed thoroughly in water, two to three times and dried in air. Assessment for the removal of soil was done as given in the next section.

4.5 Determination of reflectance of soiled and washed samples

The reflectance meter manufactured by Photovolt Corporation, Model 610, was used to obtain the reflectance values of the samples.

The Photovolt Photoelectric Reflectance Meter Model 610 is designed for measuring the diffuse reflection of surfaces as an indication of colour or degree of whiteness. The reflection meter consists of two units : (1) the instrument proper, which contains the indicating meter, the power supply and the controls, (2) the search unit, which comprises the light source and the photocell. The search unit is furnished with three tristimulus filters; amber (600-620 nm), green (520-540 nm) and blue (430-450 nm). Of these green has been recommended for reflectance measurement for detergency tests.

The green tristimulus filter is recommended since it is designed to measure luminious apparent reflectance and serves for whiteness measurement of near white materials. In the measurement of near white surfaces a test with the green tristimulus filter alone will be the only test required in detergency studies. The instrument was calibrated by using the green filter and a white tile of the instrument which reads 75 as reflectance.

The test sample was backed by a black tile and the reflectance with green filter was noted. An average of three readings was taken. Percentage soil removed was calculated by the formula given below (27) :

$$\% \text{ soil removed} = \frac{Rw - Rs}{Ro - Rs} \times 100$$

where

Ro - Reflectance of original sample

Rs - Reflectance of soiled sample

Rw - Reflectance of washed sample.

4.6 Analysis of results to get the optimum combination composition

Soil removal depends upon the solution and its characteristics and the fabric. The preliminary characteristics (see Section 4.2) were analysed and were observed in relation with soil removal.

Thus the related properties were plotted (for all the individual soaps and detergents and the combinations) to see their relationship as shown below:

a) surface tension vs conc

b) percentage wettability vs conc

c) wetting time vs conc

d) foaming power vs conc

e) emulsifying power vs conc

Per cent soil removed was calculated for the cleaning agents (individual and in combination) taking into consideration the factors involved namely fabric and soil vs cleaning agents. Analysis of results for the above was done with the help of graphs.

The latter results (that is the % soil removed) were compared with the related properties of the cleaning agents to see their effect on detergency.

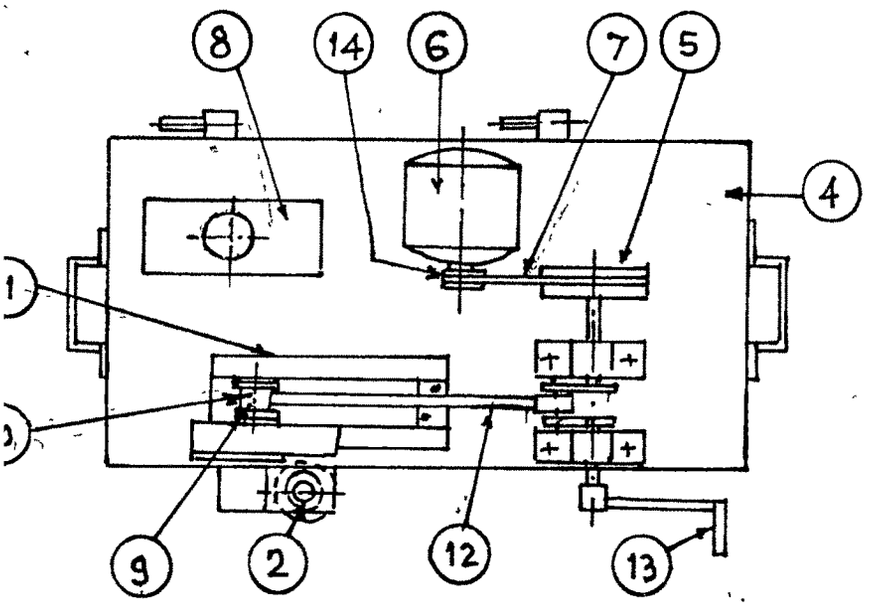
4.7 Fabrication of a small washing machine to study the applications of cleaning solutions in washing

A small washing machine with stirrer from above (to reduce cost) was fabricated, the cost being reduced by eliminating the cost of the container of the commercial washing machines and using a plastic bucket.

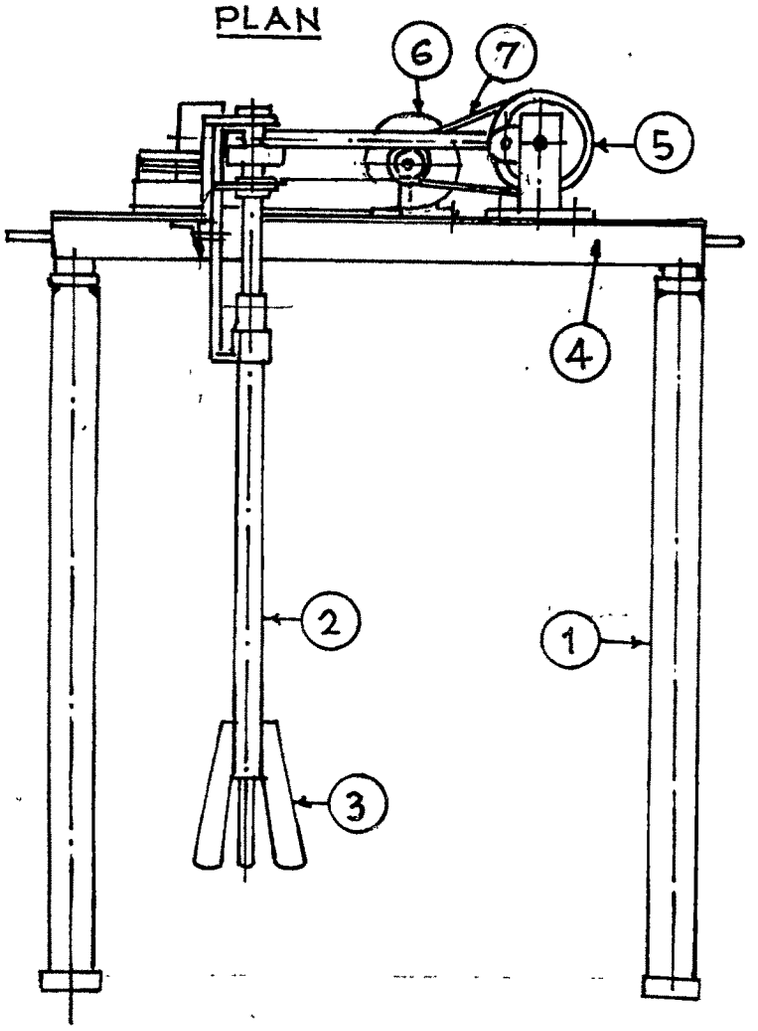
The fabrication has been illustrated in Figure 9 giving description of each part therein.

To assess the washing efficiency from application point of view, solvent soiled samples were washed in the machine. The washing was done at room temperature with 1:30 material:liquor ratio for 15 minutes; dummy load was used along with the soiled samples. After the wash cycle the samples were rinsed in water and were dried in air.

These results were compared with the results of samples washed in the Launder-Ometer.



PLAN



ELEVATION

FIG. 9 DIAGRAM OF A SMALL WASHING MACHINE

1. LEGS (STAND)
2. STIRRER ROD
3. STIRRER BLADE
4. BASE
5 BIG PULLEY
6 MOTOR
7 BELT
8 RIDER (REGULATOR)
9 RACK
10 PINION
11 RACK SLIDE
12 CONNECTING ROD
13 HANDLE FOR HAND OPERATION
14 SMALL PULLEY

Description of the small washing machine

The washing machine so designed has a stirrer to agitate the washing solution and the soiled fabrics to facilitate soil removal and efficient cleaning of the soiled garments. The complete assembly of the washing machine is fitted on a frame made out of angles with a top face of steel sheet. The frame in turn is mounted on four legs which can be fixed with a nut and bolt arrangement. These legs are detachable. The stirrer is attached to the shaft of pinion through a socket.

The washing machine consists of the following main sub-assemblies :

(a) crank assembly - the crank is made of two steel plates of 10 mm thick fixed with a shaft at the end for fitting a pulley on it. The shaft is assembled to a bracket through a ball bearing at the other end of crank a link is attached.

(b) link assembly - link assembly consists of a steel rod of 12 mm diameter with threads at both ends through which are attached crank assembly and slider.

(c) slider assembly - this assembly is made out of two steel plates of 5 mm thickness. This assembly in turns moves to and fro in guide block.

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as fabricated by Research and Development Organization, Ministry of Defence, Pune.

(d) guide block - this block consists of two channel sections fitted on the frame parallel to each other for smoother movement of the slider.

(e) rack and pinion - a rack with teeth on one of its side is attached to one of the slider plates facing outside. Pinion with similar teeth is in a bracket with brass bushing attached to the frame so that whenever slider moves pinion also rotates.

(f) stirrer - a stirrer/agitator made out of tube with three pins at one end and a cross pin at the other end. It is fixed to the shaft of pinion with a socket which is also provided with locking arrangement. An additional guide bracket is also provided for the agitator/stirrer.

(g) drive - an electric motor of 1/15 HP of Tullu make commercial model is fitted on the frame. A best and pulley arrangement can drive the crank assembly as and when supply is given to the motor.

(h) speed controller - to control the speed of motor and subsequent movement of other parts an electronic speed regulator is fitted on the frame near the motor.

The complete machine with frame and stirrer assembly can be accommodated in a wooden packing case. Four legs of the machine-frame can be housed in the lid of the case and fixed in portion

with the help of a small metal strip and screw. Details can be seen in Figure 9.

Technical Data

(a) Capacity	.. ..	1 kg of dry clothes load in 20-30 min
(b) Power required	..	1/16 HP electric motor
(c) Water required	..	approx. 20 lit.
(d) Dimensions	.. ..	515 x 290 x 650 mm
(e) Weight	.. ..	17 kg