CHAPTER IV

EXPERIMENTAL PROCEDURE

The experimental procedure has been explained under the following heads.

4.1 Preliminary data of the fabrics used.

4.2 Preparation of fabrics.

4.3 Study of a suitable catalytic system.

4.4 Preparation and application of finishes.

4.5 Determination of the physical properties.

4.6 Procedure for studying the durability of finishes.

4.7 Evaluation of appearance of fabrics after wrinkling and ironing.

4.1 Preliminary data of the fabrics used

4.1.1 Determination of thread count of fabrics[16b]

The number of ends and picks per inch in the warp and the weft were determined using the Alfred Suter counter. Average of five readings was taken. The data was also reported in the metric system.

4.1.2 Determination of weight per unit area of the fabrics[16a]

Five specimens of 5 cms x 5 cms were cut at random from the fabrics. The specimens were conditioned over saturated common salt solution at room temperature in a glass cabinet for at least twelve hours. Each conditioned sample was weighed separately on an analytical balance. An average of five readings was obtained and weight per unit area in ounces per square yard was calculated using this formula :

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

It was reported in grams per square metre, calculated from this formula:

$$\text{Weight (gm/sq.metre)} = \frac{W(\text{gms}) \times 100\ (\text{cms}) \times 100\ (\text{cms})}{5\ (\text{cms}) \times 5\ (\text{cms})}$$

4.1.3 Determination of thickness of the fabrics[16c]

Five samples of 5 cms x 5 cms were cut at random from the fabrics. The specimens were conditioned over saturated common salt solution at room temperature in glass cabinet for at least twelve hours.

The compressometer was used to determine the thickness of the fabrics. Conditioned specimens one at a time was placed on the anvil without 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 recorded from the lower dial. The pressure was then increased until the upper dial read 48 (equal to 1 lb per square inch pressure) and the lower dial reading was recorded again. The difference between the two readings (X0.001) gave the thickness of the fabric in inch. The average of five readings was taken as the fabric thickness. This was converted to centimeters and reported to the nearest 0.001cm.

4.2 **Preparation of fabrics**

4.2.1 Fabrics used: Three fabrics were used for this study. They were 100 percent cotton poplin (Fabric A), 30/70 cotton/polyester (Fabric B) and 100 percent polyester (Fabric C).

4.2.2 Scouring procedure for the fabrics

The 100 percent cotton poplin (Fabric A) was scoured in 5 g/l soap and 5 g/l soda ash at boil for 45 minutes. Cotton/polyester (Fabric B) was scoured in 2 g/1 soap and 2 g/1 soda ash at 80°C for 30 minutes and 100 percent polyester (Fabric C) was scoured in 2 g/1 soap and 2 g/1 soda ash for 30 minutes at 50-60°C. The material liquor ratio was 1:30. Fabrics were washed thoroughly in water and dried. Ironing was done to remove all creases. Samples of 12 inches x 6 inches were cut and dummy pieces of 2 inches x 6 inches were attached on one end to facilitate padding.

4.3 Study of suitable catalytic system

Table 1 gives the catalytic systems that were used to bring about the polymerization of acrylamide monomer at room temperature. On polymerization the solution turned viscous. Redox catalytic systems used included a reducing agent along with an initiator or an oxidising agent for the polymerization of acrylamide. Other agents for example trichloroacetic acid, ammonium pershulphate and epichlorohydrin which were expected to help in the polymerization of acrylamide were also added. (Time taken for the solution to turn viscous was noted.) The selection of the catalytic system used for the study was done in account with the polymerization occuring within sufficient time. It was seen that formaldehyde and hydrogen peroxide was a suitable catalytic system and addition of epichlorohydrin brought about the reaction faster. Epichlorohydrin was expected to react with cellulose. It was also seen that addition of ammonium persulphate an additional oxidising agent, was helpful in bringing the reaction faster. It was observed from Table 1 that (a redox catalytic system brought about polymerization of acrylamide while using an acid catalytic system did not,) except by the use of thichloroacetic acid and ammonium persulphate. Glyoxal-hydrogen

TABLE 1

VARIOUS CATALYTIC SYSTEMS USED

Finish mixture code	Redox catalytic system used with acrylamide monomer	Time taken for polymerization	Finish mixture code	Acid catalytic system used with acrylamide monomer	Time taken for polymerization
1	$Na_2S_2O_3 + (NH_4)_2S_2O_8$	24 hours	8	$CCl_3COOH + (NH_4)_2S_2O_8$	24 hours
2	$CH_2ClCOOH+Na_2S_2O_3+(NH_4)_2S_2O_8$	"	9	$O.CH_2.CH\ CH_2Cl + (NH_4)_2S_2O_8$	no viscosity
3	$CHOCHO + H_2O_2$	3 "	10	$O.CH_2\ CH\ CH_2Cl + CCl_3COOH$	"
4	$CHOCHO + H_2O_2+(NH_4)_2S_2O_8$	" "	11	$CH_2ClCOOH + H_2O_2$	"
5	$HCHO + H_2O_2$	20 "	12	$CCl_3COOH + H_2O_2$	"
6	$O.CH_2.CH.CH_2Cl+HCHO+H_2O_2$	16 "	13	$O.CH_2.CH.CH_2Cl+H_2O_2$	"
7	$O.CH_2.CH.CH_2Cl+HCHO+H_2O_2+(NH_4)_2S_2O_8$	12 "	14	$O.CH_2.CH.CH_2Cl+HCHO$	"
			15	$O.CH_2.CH.CH_2Cl+CHOCHO$	"
			16	$HCHO+NH_2SO_3H$	"

$Na_2S_2O_3$ = sodium thiosulphate

$(NH_4)_2S_2O_8$ = ammonium persulphate

$CH_2ClCOOH$ = chloroacetic acid

CCl_3COOH = trichloroacetic acid

H_2O_2 = hydrogen peroxide

$O.CH_2.CH.CH_2Cl$ = epichlorohydrin

HCHO = formaldehyde

CHOCHO = glyoxal

NH_2SO_3H = sulphamic acid

NOTE: For 5% of the acrylamide finish 2% of each of the above were added.

peroxide with acrylamide showed viscosity within three hours. Formaldehyde and hydrogen peroxide with acrylamide was an effective redox catalytic system. The polymerization occured faster in addition of epichlorohydrin and ammonium persulphate.

4.4 Preparation and application of finishes

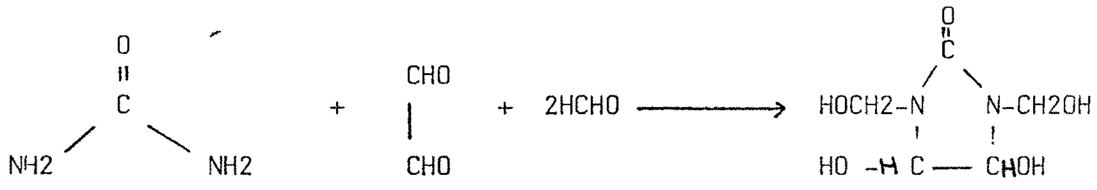
4.4.1 Finishes used : Two finishes were used in the study. They were acrylamide and Super finish EU[*], dimethylol dihydroxy ethylene urea (DMDHEU).

a) Acrylamide is thermoplastic in nature. A white crystalline solid, its melting point is 84.85°C. Acrylamide is prepared by graded hydrolysis of acrylonitrile.

$$CH_2=CH-CN+H_2O \longrightarrow CH_2=CH-CO-NH_2$$

It is reactive, wet and dry crease recovery are obtained by its application. Acrylamide has the capacity to undergo addition polymerization to form a linear polymer thousands of unit long.[38]

b) The characteristics of Superfinish EU, as given by the manufacturer is that it is a reactive thermosetting resin having the chemical nature of dimethylol dihydroxy ethylene urea. It is a pale yellow liquid and imparts shrink proof crease resistance finish to cellulose and cellulose/ synthetic blends. It is prepared by reacting urea, glyoxal and formaldehyde.[63]



Urea glyoxal formaldehyde DMDHEU

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[*] Manufactured by Texchem, Dr.Annie Besant Road, Bombay 400 018.

4.4.2 Preparation of the finish

It was observed from Table 1 that epichlorohydrin along with formaldehyde-hydrogen peroxide catalytic system brought about the polymerization faster. It was also expected to crosslink with cellulose therefore it was used in the study. The application of the finish was varied to obtain maximum reaction between the fibre and finish.

The following recipes of the finish at 5% and 10% concentration were padded on the fabrics.

Part - I

Finish Recipe	conc. 5%	% Wt.	conc. 10%	% wt.
1) t_1				
Acrylamide	5 g	5	10 g	10
Epichlorohydrin	2 ml	2	4 ml	4
Formaldehyde (40%)	2 ml	0.8	4 ml	1.6
Hydrogen peroxide (20vol)	2 ml	0.24	4 ml	0.48
Teepol 1 g/l	94 ml	0.1	88 ml	0.1
Total	100 ml	8.14	100 ml	16.18
2) t_2				
1st Step:				
Acrylamide	5 g	5	10 g	10
Epichlorohydrin	2 ml	2	4 ml	4
Formaldehyde (40%)	2 ml	0.8	4 ml	1.6
Teepol 1 g/l	96 ml	0.1	92 ml	0.1
Total	100 ml	7.90	100 ml	15.70

Finish Recipe	conc. 5%	% Wt.	conc. 10%	% Wt.
2nd Step:				
Hydrogen peroxide (20vol)	2 ml	0.24	4 ml	0.48
Sodium carbonate	500 mg	0.5	1 g	1.0
Teepol 1 g/l	98 ml	0.1	96 ml	0.1
Total	100 ml	0.84	100 ml	1.58
3) t_{3a} (pH8)				
1st step: Pretreatment				
Epichlorohydrin	2 ml	2	4 ml	4
Formaldehyde (40%)	2 ml	0.8	4 ml	1.6
Teepol 1 g/l	96 ml	0.1	92 ml	0.1
Total	100 ml	2.9	100 ml	5.7
2nd Step:				
Acrylamide	5 g	5	10	10
Formaldehyde (40%)	2 ml	0.8	4 ml	1.6
Hydrogen peroxide (20vol)	2 ml	0.24	4 ml	0.48
Teepol 1 g/l	96 ml	0.1	92 ml	0.1
Total	100 ml	6.14	100 ml	12.18
t_{3b} (pH3)				
1st Step: Pretreatment				
Epichlorohydrin	2 ml	2	4 ml	4
Formaldehyde (40%)	2 ml	0.8	4 ml	1.6
Teepol 1 g/l	96 ml	0.1	92 ml	0.1
Total	100 ml	2.9	100 ml	5.7

Finish Recipe	conc. 5%	% Wt.	conc. 10%	% Wt.
2nd Step:				
Acrylamide	5 g	5	10	10
Formaldehyde (40%)	2 ml	0.8	4 ml	1.6
Hydrogen peroxide (20vol)	2 ml	0.24	4 ml	0.48
Ammonium persulphate	1 g	1.0	2 g	2.0
Teepol 1 g/l	96 ml	0.1	92 ml	0.1
Total	100 ml	7.14	100 ml	14.18
t_{3c} (pH6)				
1st Step: Pretreatment				
Epichlorohydrin	2 ml	2	4 ml	4
Formaldehyde (40%)	2 ml	0.8	4 ml	1.6
Teepol 1 g/l	95.5ml	0.1	91.5ml	0.1
Acetic acid	0.5ml	0.5	0.5ml	0.5
Total	100 ml	3.4	100 ml	6.2
2nd Step:				
Acrylamide	5 g	5	10 g	10
Formaldehyde (40%)	2 ml	0.8	4 ml	1.6
Hydrogen peroxide (20Vol)	2 ml	0.24	4 ml	0.48
Teepol 1 g/l	95.5ml	0.1	91.5ml	0.1
Acetic acid	0.5ml	0.5 ml	0.5ml	0.5
Total	100 ml	6.64	100 m	12.68

Finish Recipe	Conc. 5%	% Wt.	Conc. 10%	% Wt.
t_{3d} (pH10)				
1st Step: Pretreatment				
Epichlorohydrin	2 ml	2.0	4 ml	4.0
Formaldehyde (40%)	2 ml	0.8	4 ml	1.6
Teepol 1 g/l	94 ml	0.1	90 ml	0.1
Ammonia (25%)	2 ml	0.5	2 ml	0.5
Total	100 ml	3.4	100 ml	6.2
2nd Step:				
Acrylamide	5 g	5	10 g	10
Formaldehyde (40%)	2 ml	0.8	4 ml	1.6
Hydrogen peroxide (20vol)	2 ml	0.24	4 ml	0.48
Teepol 1 g/l	94 ml	0.1	90 ml	0.1
Ammonia (25%)	2 ml	0.5	2 ml	0.5
Total	100 ml	6.64	100 ml	12.68

PART - II

Finish Recipe	conc. 5%	% Wt.	conc. 10%	% Wt.
Finish I				
1st Step: Pretreatment				
Epichlorohydrin	2 ml	2.0	4 ml	4.0
Formaldehyde (40%)	2 ml	0.8	4 ml	1.6
Teepol 1 g/l	96 ml	0.1	92 ml	0.1
Total	100 ml	2.9	100 ml	5.7
2nd Step:				
Acrylamide	5 g	5	10 g	10
Formaldehyde (40%)	2 ml	0.8	4 ml	1.6
Hydrogen peroxide (20vol)	2 ml	0.24	4 ml	0.48
Ammonium persulphate	1 g	1.0	2 g	2.0
Teepol 1 g/l	96 ml	0.1	92 ml	0.1
Total	100 ml	7.14	100 ml	14.18
Finish II				
1st Step: Pretreatment				
Epichlorohydrin	2 ml	2	4 ml	4
Formaldehyde (40%)	2 ml	0.8	4 ml	1.6
Teepol 1 g/l	96 ml	0.1	92 ml	0.1
Total	100 ml	2.9	100 ml	5.7

Finish Recipe	Conc. 5%	% wt.	Conc. 10%	% wt.
2nd Step:				
Acrylamide	2.5g	2.5	5 g	5
DMDHEU	2.5ml	1.25	5 ml	2.5
Formaldehyde (40%)	1 ml	0.4	2 ml	0.8
Hydrogen peroxide (20vol)	1 ml	0.12	2 ml	0.24
Ammonium persulphate	1 g	1.0	2 g	2.0
Teepol 1 g/l	95.5ml	0.1	91 ml	0.1
Total	100 ml	5.37	100 ml	10.64
Finish III				
DMDHEU	5 ml	2.5	10ml	5.0
Ammonium persulphate	1 g	1.0	2 g	2.0
Teepol 1 g/l	95	0.1	90	0.1
Total	100ml	3.6	100ml	7.1

PART III

	Finish Recipe	Conc. 5%	% wt.	Conc. 10%	% wt.
	Finish Ix				
	Precondensate				
1)	Acrylamide	5g	5	10g	10
2)	Epichlorohydin	2ml	2	4ml	4
3)	Formaldehyde (40%)	2ml	0.8	4ml	1.6
4)	Teepol (1gle)	92ml	0.1	84ml	0.1
5)	Ammonia (25% as needed to get pH8)	<1ml	0.12	<1ml	0.12
6)	Formaldehyde (40%)	2ml	0.8	4ml	1.6
7)	Hydrogen peroxide(20vol)	2ml	0.24	4ml	0.48
8)	Ammonium persulphate	1g	1.0	2g	1.0
	Total	100ml	10.06	100ml	18.9

1 to 5 were mixed first, 6,7, and 8 were added just before padding (formaldehyde was added in two parts as above).

	Finish Recipe	Conc. 5%	% wt.	Conc 10%	% wt.
	Finish IIx				
	Precondensate				
1)	Acrylamide	2.5g	2.5	5g	5
2)	Epichlorohydin	1ml	1	2ml	2
3)	Formaldehyde (40%)	1ml	0.4	2ml	0.8
4)	Teepol (1g/1)	46ml	0.1	42ml	0.1
5)	Ammonia (25% as needed to get PH8)	<1ml	0.12	<1ml	0.12
6)	Formaldehyde (40%)	1ml	0.4	2ml	0.8
7)	Hydrogen peroxide(20vol)	1ml	0.12	2ml	0.24
8)	DMDHEU (50%)	2.5ml	1.25	5ml	2.5
9)	Ammonium persulphae	1g	1.0	2g	2.0
10)	Teepol (1g/1)	47.5ml	0.1	45ml	0.1
	Total	100ml	6.99	100ml	13.66

1 to 5 were mixed first, 6,7,8,9 and 10 were added just before padding (formaldehyde and teepol were added in two parts as above)

4.4.3 Procedure for application of finish

PART I

The application of acrylamide was varied to bring about a reaction between the fibre and finish.

1. With t_1, one set of fabrics were padded with varying concentration of acrylamide finish and air dried.
2. With t_2, application of hydrogen peroxide only (made alkaline with sodium carbonate) to accelerate the reaction as second step.
3. With t_3, a pretreatment of epichlorohydrin and formaldehyde was given to obtain the reaction via the two, followed by aplication of acrylamide finish as second step.

a) With formaldehyde and hydrogen peroxide (simple redox system), pH = 8.

b) With formaldehyde, hydrogen peroxide and ammonium persulphate (mixed redox system), pH = 3.

c) With formaldehyde and hydrogen peroxide (simple redox system) made acidic with acetic acid, pH = 6.

d) With formaldehyde and hydrogen peroxide (simple redox system) made alkaline with ammonia, pH = 10.

The pH of the pretreatment step in 'c' and 'd' was also changed to acidic (pH=6) and alkaline (pH=10) respectively. Variation in the pH was done to see its effect on the reaction between the fibre and finish.

Part II

4) The application of acrylamide finish (Finish I) in two steps:

1st Step: Pretreatment with epichlorohydrin and formaldehyde

2nd Step: Acrylamide finish with mixed redox system (formaldehyde, hydrogen peroxide and ammonium persulphate).

5) A combination of acrylamide and DMDHEU (Finish II) was padded on the fabrics in two steps:

1st Step: Pretreatment with epichlorohydrin and formaldehyde

2nd Step: 1:1 combination (wet basis) of acrylamide and DMDHEU.

6) DMDHEU (Finish III) was padded on the fabrics.

Part III

7) The precondensate (Finish Ix) was padded on the fabrics

8) Combination of acrylamide precondensate and DMDHEU (Finish IIx) was padded on the fabrics

The prepared samples of 12" x 6" were padded with the solutions on a padding mangle with appropriate weight to get the approximate percent pick-up:

1500 grams for 100 percent cotton fabric (A)
500 grams for cotton/polyester fabric (B)
500 grams for 100 percent polyester fabric (C)

The percent pick-up for cotton fabric (A) was 110% and 100% for cotton/polyester fabric (B) and polyester fabric (C). Samples were first dried at room temperature and later cured at 130°C for 5 minutes.

4.5 Determination of the physical properties

4.5.1 Determination of wrinkle recovery[4b]

Wrinkle recovery was determined on Monsanto wrinkle recovery tester.

Six samples measuring 4 cm x 1.5 cm were cut in the warp direction from the fabric samples subjected to different treatments. The samples were conditioned prior to testing.

Using tweezers, a test specimen was placed between the leaves of the specimen holder with one end flush with the longer metal strip. 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 thumb nail. 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 of and parallel to the longer metal strip of the holder.

The press holder combination was inverted on a table top with the small platform upward and a load of 500 grams was applied to the platform. Load was removed exactly after five minutes and 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 was removed from the press jaw, after which the specimen holder was properly aligned on the mounting shelf. The crease was lined up with a line 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 effect, it was necessary to keep the dangling specimen leg aligned with the vertical guide line, during five minutes recovery period.

After five minutes of recovery, final adjustment of the dangling leg to the vertical guide line was made, and the wrinkle recovery value was read from the protractor scale by means of the vernier. Average of six readings was taken as the recovery of the fabrics.

4.5.2 Determination of Stiffness[6]

For measuring the stiffness, bending cantilever method was used as given below:

The conditioned samples of size 15 cms x 2.5 cms (the longer being the warp direction) were tested. The sample strip was kept on the platform and moved slowly. When the sample touched the slope at 45°, then the bending length was measured on the platform of the instrument. Average of five reading was taken.

4.5.3 Determination of tensile strength and elongation[16d]

The tensile strength and elongation of the fabrics with varying levels of treatments were determined on the Scott tester. Sample size of 6 inches x 1 inch (15 cms x 2.5 cms) were cut in the warp direction. The samples were ravelled from either side to measure 6 inch x 0.8 inch (15cms x 2.0cms). The samples were conditioned in a standard atmosphere. The distance between the two jaws was 3 inches (7.5 cms). The specimen was mounted in the two jaws. The machine was started, the lower jaw was pulled downward till the sample broke. The breaking strength and elongation were noted from the graph. An average of five readings was taken. The percentage elongation at breaking point was calculated from the formula:

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

Y = elongation obtained from the graph

X = original length of specimen

4.6 Procedure for studying the durability of the finish[4a]

Durability of finishes was assessed (of samples dried at room temperature and samples cured at 130°C for 5 minutes) by washing, using Method IIA of AATCC 1965. Material: liquor ratio was 1:20 and 2 gm/l soap was used. Samples were kept in the solution with occasional stirring for 30 minutes maintaining the temperature at 55°C - 60°C. They were then washed under running water, dried and conditioned.

1) % Weight loss was calculated.

2) residual wrinkle recovery and stiffness were assessed.

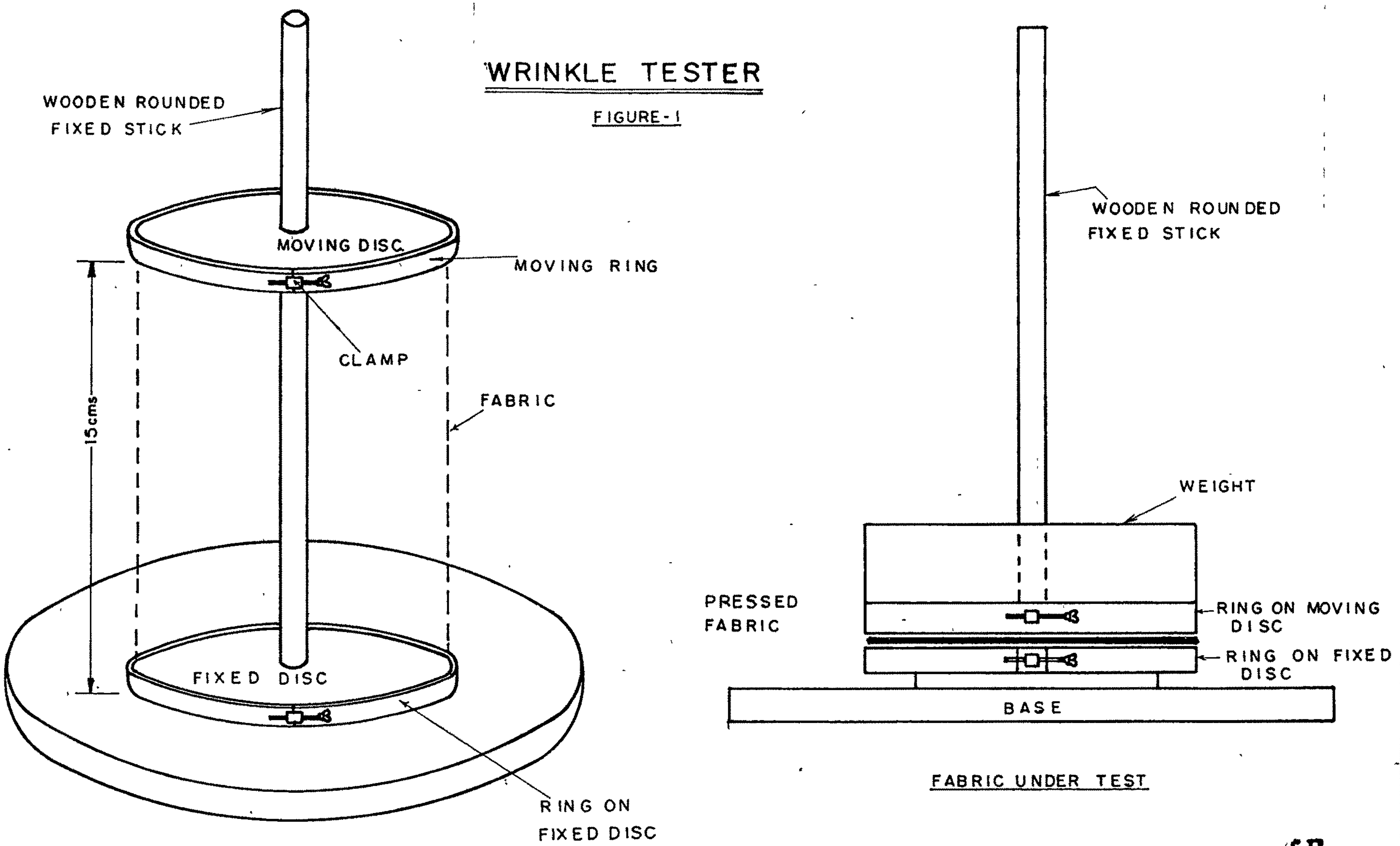
From these results durability of the finish was ascertained in general.

4.7 Evaluation of appearance of fabrics after wrinkling and ironing[4c]

The principle of AATCC test method 128-1969 was the basis of the experiment. The apparatus made by Phadke[51] shown in Figure 1 was used. Three test specimens of size 28 cms x 15 cms with long dimension running in the direction of the warp were cut and ironed at nylon setting and then conditioned in a standard atmosphere over a saturated solution of common salt. The top wooden disc of the tester was raised and the long edge of the specimen (28 cm) was wrapped around the top disc with the face side of the specimen on the outside. The specimen was clamped with a wooden ring with screw. The other edge of the specimen was similarly fixed around the botton disc. The specimen was adjusted by pulling at

WRINKLE TESTER

FIGURE-1



FABRIC MOUNTED FOR TESTING

FABRIC UNDER TEST

the bottom edge, so that it was smooth without sagging between the top and the bottom discs. The top disc was gently lowered with one hand till it came to rest. The two discs were then covered with a cut tin box and a total of 3,500 grams weight was placed on the top of the box. After 20 minutes the weights and the box was removed. The top disc was raised so as not to distort any induced wrinkles.

The specimen was then hung on the screen with a hanger having adjustable clips. The background of the screen was gray and the wrinkles were observed under an overhead fluorescent lighting at an angle of 15°. The total height from the floor to the light was eight feet and the distance of the sample from the floor was five feet.

The observer stood directly in front of the test specimen, four feet away from the screen. For comparison, two photographs of three dimensional replicas were hung on each side of the test specimen to facilitate comparative rating. Equal portion of the photograph of three dimensional replica to the fabric specimen size was only exposed for comparison, the remaining part being covered with a gray paper sheet. These photographs were changed by the investigator as desired by the observer. Each test specimen was independently evaluated by three raters and assigned the numbers of the photograph of replica which most nearly matches the appearance of the test specimen. Test specimens were again rated by the three observers after 24 hours. To see any influence of the weight of the iron alone, each test specimen was ironed flat with a cold iron for 10 seconds on both sides of the fabric. The test specimens were then again rated by three observers. To see the ease of ironing, each test specimen was ironed flat with iron at nylon setting using the same procedure as above. After ironing, the test specimen were rated by the three observers. The fabric smoothness ratings were:

No.1 Rating was equivalent to standard 1 and represented the poorest appearance and poorest retention of original appearance.

No.2 Rating was next to No.1.

No.3 Rating was next to No.2.

No.4 Rating was next to No.3.

No.5 Rating was equivalent to standard 5 and represented the smoothest appearance and the best retention of original appearance.

The analysis of the results was done graphically. The effect of each finish at varying concentrations was shown for each fabric.

On the basis of the change in the physical properties, durability and appearance rating of fabrics, the most suitable finish for wash and wear was reported.