THE EFFECTS OF LAUNDRY VARIABLES ON THE, FLAME RETARDANCY OF A CORDELAN/COTTON, SLEEPWEAR KNIT

by

1,10

SELMA SUE FENT

226-5600

B. S., Kansas State University, 1970

A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Clothing, Textiles and Interior Design

KANSAS STATE UNIVERSITY Manhattan, Kansas

1973

Approved by:

Major Professor

LD

Chapter	F	age
	CALCIUM CONTENT DETERMINATION	16
	PHOTOMICROGRAPHS	17
	STATISTICAL ANALYSIS	18
4. R	ESULTS	19
	VERTICAL FLAME TEST	19
	Residual Flame Time	20
	Char Length	20
	CALCIUM CONTENT	24
	STATISTICAL ANALYSIS	24
	Residual Flame Time	24
	Char Length	30
	Calcium Content	31
	PHOTOMICROGRAPHS	33
5. 0	ONCLUSIONS	34
LITERATU	RE CITED	36
APPENDIX	ES	38
A. Cu	tting Plan for Test Fabric Specimens	39
B. Se	lected Scanning Electron Photomicrographs	41

LIST OF TABLES

Table		Page
1.	Examples of Flame Retardant Finishes	6
2.	Examples of Modified Fibers	7
3.	Examples of Inherently Flame Retardant Fibers	7

LIST OF FIGURES

Figure		Page
1.	Average Residual Flame Times of Specimens Washed with "Dash" Detergent 0, 1, 4, 9, 16, 36, 50 and 55 Times, with or without Bleach at 150 ppm or 300 ppm Water Hardness	21
2.	Average Residual Flame Times of Specimens Washed with "Sears" Detergent 0, 1, 4, 9, 16, 25, 36, 50 and 55 Times, with or without Bleach at 150 ppm or 300 ppm Water Hardness	22
3.	Average Residual Flame Times of Specimens Washed with "Cold Water All" Detergent 0, 1, 4, 9, 16, 25, 36, 50 and 55 Times, with or without Bleach at 150 ppm or 300 ppm Water Hardness	23
4.	Average Char Lengths of Specimens Washed with "Dash" Detergent 0, 1, 4, 9, 16, 25, 36, 50 and 55 Times, with or without Bleach at 150 ppm or 300 ppm Water Hardness	25
5.	Average Char Lengths of Specimens Washed with "Sears" Detergent 0, 1, 4, 9, 16, 25, 36, 50 and 55 Times with or without Bleach at 150 ppm or 300 ppm Water Hardness	26
6.	Average Char Lengths of Specimens Washed with "Cold Water All" Detergent 0, 1, 4, 9, 16, 25, 36, 50 and 55 Times, with or without Bleach at 150 ppm or 300 ppm Water Hardness	27
7.	Average Calcium Content of Swatches Washed with "Dash" Detergent 0, 16, 25 and 50 Times, with and without Bleach at 150 ppm and 300 ppm Water Hardness	28
8.	Average Calcium Content of Swatches Washed with "Cold Water All" Detergent 0, 16, 25 or 50 Times, with and without Bleach at 150 ppm and 300 ppm Water Hardness	28
9.	Average Calcium Content of Swatches Washed with "Sears" Detergent 0, 4, 9, 16, 25, 36, 50 and 55 Times, with and without Bleach at 150 ppm and 300 ppm Water Hardness	29

ACKNOWLEDGMENTS

Sincere appreciation is expressed to Dr. Wayne L. St. John, Department of Clothing, Textiles and Interior Design for personal encouragement and guidance in directing this study and in preparation of this thesis.

The author wishes to express thanks to committee members, Dr. Jessie A. Warden, Head of the Department of Clothing, Textiles and Interior Design and Patty J. Annis, Department of Family Economics for their interest and constructive suggestions.

Further appreciation is extended to Dr. Ray A. Waller and Richard E. Thomas for help with statistical analysis; to Paul D. Enos for scanning electron photomicrographs; and to Daniel K. Thompson, my family and friends for encouragement, understanding and moral support.

Chapter 1

INTRODUCTION

Each year an estimated 150,000 to 200,000 persons suffer injuries involving burning textiles, and 3000 to 5000 die as a result. Aside from being physically and psychologically devastating, burn injuries can be expensive. Annually, treatment costs and earning losses are estimated at over \$250 million. An investigation conducted by the Department of Commerce, The National Bureau of Standards and the Food and Drug Administration showed that the very young (ages 0-5) and the old (ages 66+) are most seriously affected by injuries from burning textiles (2, 3, 10, 17, 24).

Sleepwear garments are frequently involved in fires resulting in burn injuries to young children. Males aged 0-5 received almost four times and females in this age group 1.6 times the number of sleepwear burn injuries expected on the basis of their percentage of the total population (2, 3, 10, 24, 25). Such findings have prompted Federal legislation concerning the flammability of textiles. As of July 29, 1973 all ready-to-wear sleepwear up to size 6X and fabrics intended or promoted for such use must pass test criteria set forth by the Standard for the Flammability of Children's Sleepwear (DOC FF 3-71) (8). For one group of textile consumers more prone to textile burn injuries than other groups, this standard gives protection beyond that provided by the Flammable Fabrics Act (1953) and its amendment (1967).

The textile industry is currently involved in research and development to supply fabrics that will meet government standards for flammability. At the same time it is concerned with providing such consumer demanded qualities as long service life, strength, attractive appearance, handle, comfort,

absorbency, launderability and competitive cost (2, 7, 10).

One of the many problems the industry has encountered in developing and testing flame retardant fabrics and finishes involves laundry treatments. Pacheco and Carfagno (23) found that use of a carbonate detergent led to a build-up of calcium carbonate when used in hard water. They also found that the use of soap led to a build-up of calcium stearate. These build-ups increased the flammability of cotton flannelette treated with a flame retardant finish. Pacheco and Carfagno found that such deposits do not build up when a high phosphate detergent is used. Similar results were reported by Perkins, Drake and Reeves (24).

In other areas phosphate levels in detergents are controlled (23). It appears that state and local laws governing types of laundry products to be used are in conflict with Federal standards in those instances where build-up due to use of a sanctioned detergent renders children's sleepwear flammable after a few washings.

Research is continuing in the textile industry to find fabrics that will pass the test criteria for Federal Flammability Standards when the fabrics are washed in hard water with locally available detergents. This study was conducted to investigate the effects of various laundry treatments on the flammability of a 87 1/2% Cordelan (polyvinyl chloride-polyvinyl alcohol or PVC-PVA)/12 1/2% cotton sleepwear knit.

This inquiry may serve as a basis for further study of the effects of laundry variables on the flame retardancy of this or other flame retardant fabrics. Objectives of this study were:

- 1. To investigate flammability of a Cordelan/cotton blend sleepwear knit at nine wash cycle levels (in a series of fifty-five launderings) under twelve different laundry conditions.
- 2. To investigate calcium deposition on the above mentioned fabric as a result of washing (through calcium content analysis and exploratory scanning electron photomicrographs) as a possible cause of loss in the flame retardant quality.

Chapter 2

REVIEW OF LITERATURE

Most fibers used in textiles are organic in character. Most organic compounds are capable of supporting combustion if a sufficient temperature is attained and adequate oxygen is available. When a fabric is ignited the organic fibers decompose and frequently liberate decomposition products which are also combustible. The fabric bursts into flames. Slow oxidation of the remaining carbon char is achieved by flameless combustion; this process consumes the fabric, liberates additional heat much more intense than that of the flame and is evidenced by a glowing action.

In developing flame retardant fabrics for clothing use, factors that should be examined are ease of ignition, burning rate, volume and temperature of flame and noxious vapors evolved during combustion, total heat produced, ignition of the back of the fabric resulting in actual contact of the flame with the skin surface, and ignition of adjacent layers of fabric. These factors depend on the chemical and physical nature of the fiber and fabric; yarm size, twist and method of yarn construction; type of fabric construction; chemical additives and techniques used in fabric finishing (13). No flame resistant fiber or flame retardant fabric will provide a self-extinguishing garment under all conditions. Three approaches to producing flame retardant fabrics have been mentioned (2).

- 1. Application of flame retardant finishes to the fabric.
- Modification of existing fibers to make them flame retardant.
- 3. Use of inherently flame retardant fibers.

FLAME RETARDANT FINISHES

A widely practiced approach to flame retardant fabrics is that of applying a chemical flame retardant to cotton (11). The primary role of a chemical flame retardant is to alter the process of decomposition so that, consequently, a smaller amount of flammable gas is produced (13). The ideal finish should be inexpensive and applicable with conventional finishing equipment. It should not affect strength, color or aesthetic qualities of the fabric, and it should be durable to various laundry and dry cleaning procedures. In addition the finish should be stable to ultraviolet light and heat and be non-toxic and non-allergenic (2).

Flame retardant finishes offer a number of problems. They may decompose to produce toxic gases causing a health hazard greater than fire (13). They require proper application and care to assure maximum efficiency and durability. Pressing reduces effectiveness of the flame retardant. This appears to be due, in part, to degradation of the finish to water soluble products that leach out during washing. A build-up of foreign matter also reduces flame retardancy (9, 22). Frequently, manufacturers' care suggestions proscribe the use of soap, bleach and low or non-phosphate detergent as well as the acid sours typically used in commercial laundering (11).

Currently, Pyrovatex and THPC are the leading durable flame retardant finishes commercially available for use on cottons (2). These and some other flame retardant finishes are listed in the following table (12). Jancik (16) found that cotton finished with THPC burned less often and more slowly than those finished with APO-THPC or THPOH-NH₃ and that finishes were more effective on 100% cotton on 70/30 or 50/50 blends with polyester.

Table 1
Examples of Flame Retardant Finishes

Trade Name	Company	Active Chemicals	Applications
Fammetin	Dr. Quehl	Organic Halogens	varied
Fire Retardant	Fancourt	Boro-Phosphate	cellulosics
MCC/100	Monsanto	Proprietary Phosphorous compound	cellulosics
Pyrovatex	Ciba-Geigy	Organic Phosphate	cellulosics
Pyroset	American Cyanamid	Phosphorus, Nitrogen	cellulosics
THPC	Hooker	Phosphonium Chloride	cotton, wool
APO	Dow Chemical	Tris (1-aziridiny1) phosphine oxide	cellulosics

MODIFIED FIBERS

The man-made cellulosic fibers, acetate and rayon lend themselves readily to addition of chemical flame retardants due to the methods of spinning used. Of the major synthetic fibers (nylon, acrylic, polyester and polypropylene) only acrylic can be readily modified. Since chemicals are added to the spinning solution, the flame retardant is incorporated throughout each fiber. Examples of modified cellulosic fibers are listed in the following table (2):

Table 2
Examples of Modified Fibers

Trade Name	Company	Fiber
Acele	Du Pont	acetate
Estron	Eastman	acetate
Flame Retardant Arnel	Celanese	triacetate
PFR	FMC	rayon
Sayfr	FMC	acetate

INHERENTLY FLAME RETARDANT FIBERS

Research in developing inherently flame retardant fibers is increasing. Some examples of fibers that are inherently flame retardant are listed in the following table (3):

Table 3

Examples of Inherently Flame Retardant Fibers

Trade Name	Company	Fiber
Cordelan	Kohjin	<pre>polyviny1 chloride - polyviny1 alcohol</pre>
Durette	Monsanto	phenolformaldehyde cross-linked polymer
Dyne1	Union Carbide	acrylonitrile and vinyl chloride

Table 3 (Continued)

Nomex	Du Pont	aromatic nylon
PBI	Celanese	poly-m-phenylene dibenzimidazole
SEF	Monsanto	modacrylic
Verel	Eastman Kodak	acrylonitrile vinylidene chloride
		6

Many inherently flame retardant fibers produce fabrics lacking in function and aesthetics (such as dyeability, good moisture regain, handle and drapeability) demanded by the apparel markets (4). Attempts have been made to produce desired fabric characteristics by blending inherently flame retardant fibers with natural fibers. However, these blends tend to burn rapidly because the natural fibers keep the fabric from shrinking away from the flame.

Generally, fabrics made from blends have a lower flame retardancy than do fabrics of one fiber (2, 6, 28).

LAUNDRY VARIABLES

Although DOC FF 3-71 requires use of a high phosphate detergent for test laundering sleepwear fabrics, other types of laundry products are used in home care of children's sleepwear, particularly in localities that have executed legislation restricting type of laundry products to be sold to consumers.

Phosphate detergents have been banned in Chicago, Illinois; Dade County, Florida and Erie County, New York. In Suffolk County, New York, detergents must be soap based. The states of New York, Indiana, Maine and Connecticut have limited phosphate to 8.7% (23).

CORDELAN

Cordelan, formerly known as Cordela, is the trade name of a biconstituent synthetic fiber produced by an emulsion spinning process by Kohjin Co., Ltd. of Japan. This fiber is composed mainly of polyvinyl chloride and polyvinyl alcohol with a small amount of vinylchloride grafted polyvinyl alcohol.

The polymers from which Cordelan is made and the method of spinning were developed by Professor Seizo Okamurs, Kyota University and commercialized by Kohjin Co., Ltd. in November 1967 (18). Cordelan was first marketed in the United States by Ameritex Corporation in early 1972 (27). Currently, it is classified in the U.S. under the category of matrix (biconstituent) fiber: 50% vinal (polyvinyl alcohol)/50% vinyon (polyvinyl chloride) (19).

Cordelan's outstanding characteristics are good flame retardancy, ready dyeability, high abrasion to chemicals and solvents, high abrasion resistance and a useful moisture regain (3.2%) which minimizes static (7).

Chapter 3

METHOD OF PROCEDURE

FABRIC

Fabric selected for testing was a 87 1/2% Cordelan/ 12 1/2% cotton sleepwear knit. The fabric was a light yellow color, an 11 cut flat knit jersey type, weighed 3.5 ounces per square yard + 5% and had an appearance and handle similar to T-shirt fabric.

SAMPLING PLAN

The test fabric was cut into 72 small swatches (12" in the weft direction x 36" in the warp direction) and 25 large swatches (12" in the weft direction x 54" in the warp direction). Small swatches provided two samples of five specimens each. The large swatches provided three samples of five specimens each (see Figure 1 in Appendix A) DOC FF 3-71 requires that at least five specimens from each sample be subjected to the vertical flame test described therein. The supplier of the fabric, J. C. Penney Co., requested that fifteen specimens (three samples) be tested after 0, 25 and 50 washings under each condition. Ten specimens (two samples) were tested after other wash cycle levels.

From each sample, three specimens were cut in one fabric direction and two specimens were cut in the other fabric direction as specified by DOC FF 3-71.

LAUNDRY EQUIPMENT

Test fabric swatches were washed in a Kenmore Automatic Washer model 500 (setting B: ten minute warm wash, warm rinse, normal agitation and spin).

AATCC Test Method 124, Appearance of Durable Press Fabrics after Repeated Home Launderings, as specified by DOC FF 3-71 was used (1). Method 124 specifies the Kenmore Automatic Washer model 600 as standard and accepts any other washer that provides comparable washing conditions (agitation speed 70 \pm 5 cycles per minute, normal wash time: 12 minutes, wash temperature $105 \pm 5^{\circ}$ F $(41 \pm 3^{\circ}$ C), spin speed: 500-510 rpm, final spin 4 minutes and rinse temperature: $105 \pm 5^{\circ}$ F. The Kenmore Automatic Washer model 500 was used because it was available, and it meets the requirements of AATCC Test Method 124 with the exception of the wash time which is ten minutes instead of twelve minutes.

Fabrics may be either line or tumble dried according to AATCC Test
Method 124. The tumble method was selected due to its convenience. Method
124 specifies the Kenmore Automatic Dryer model 600 as standard and accepts
any other dryer with a controlled exhaust temperature of 140 to 160°F (60 to
71°C) and a cooling period while tumbling of five minutes at the end of the
drying cycle. A Frigidaire Automatic Dryer model DIAF-61 meeting these
requirements was used.

NUMBER OF WASHINGS

Specimens from test samples were tested for flammability after 0, 1, 4, 9, 16, 25, 36, 50 and 55 washings. Tests at levels 0, 25 and 50 were requested by the supplier of the fabric. They correspond with levels tested in the Pacheco and Carfagno study (23). Fabric used in children's sleepwear must pass DOC FF 3-71 test criteria after 50 washings. Level one was tested to determine if an appreciable difference in flammability occurred after one laundering. Levels four and nine were chosen as these levels are close to those at which fabrics laundered in soap and carbonate detergent, respectively,

lost flame retardancy in the Pacheco and Carfagno study. Level fifty-five was selected to determine if those test fabrics passing at fifty launderings were on the verge of failing. Levels sixteen and thirty-six were chosen arbitrarily to supply intermediary data.

LAUNDRY PRODUCT SELECTION

Three types of detergents were used to wash test fabric swatches:

- 1) a high phosphate detergent (Dash)
- 2) a carbonate detergent (Sears)
- 3) a citrate detergent (Cold Water All)

A high phosphate detergent was used as specified by DOC FF 3-71. "Dash" was the phosphate detergent selected because of its low sudsing property. A suds build-up would probably occur in the absence of soil during repeated washings with a high sudsing product.

A carbonate detergent, "Sears," was selected because it is a commonly used alternative to a high phosphate product. This type was also used in the Pacheco and Carfagno work.

A citrate detergent is another alternative to a high phosphate product. "Cold Water All" was an example of a specially formulated liquid detergent, produced for sale in areas restricting or prohibiting phosphates in detergents.

Bleach was used in six of the twelve laundry conditions to determine if this factor affects the flame retardant properties of this particular fabric.

WATER HARDNESS LEVEL SELECTION

Two water hardness levels were chosen, 150 ppm and 300 ppm,

representing hard and very hard water. A water hardness level of 150 ppm is close to that used in the Pacheco and Carfagno study (155 ppm). A level of 300 ppm was chosen to determine if a higher water hardness level had a more deleterious effect on the flame retardancy of the test fabric. About twenty percent of U.S. households have water of hardness greater than 150 ppm, and about two percent of U.S. households have water of hardness greater than 300 ppm (26). However, Kansas has a higher incidence of hard water than does the U.S. as a whole. In Kansas eighty percent of cities provide water with hardness greater than 150 ppm and forty percent have water of hardness greater than 300 ppm (5).

Water available for laundering was presoftened as it came from the tap. Therefore, a water hardness concentrate was added to bring water used in washing to specified hardness (150 ppm or 300 ppm). The water hardness concentrate was prepared from:

441 gms. Calcium Chloride, Dihydrate (CaCl₂·2H₂0)

203 gms. Magnesium Chloride (MgCl₂·6H₂0)

1000 ml. Distilled Water

At the beginning of each day of laundering the hardness of the water was measured by EDTA titration. Hardness concentrate was added to increase water hardness to the desired level. Generally 25 ml. and 50 ml. were needed to attain 150 ppm and 300 ppm, respectively.

LAUNDRY CONDITIONS

Wash loads consisting, initially, of eight test fabric units and filler cloth to equal four pounds were subjected to the twelve following laundry conditions:

- 1) Dash (high phosphate), 90 gms.; 150 ppm; no bleach
- 2) Dash (high phosphate), 90 gms.; 300 ppm; no bleach
- 3) Dash (high phosphate), 90 gms.; 150 ppm; bleach, 1 cup
- 4) Dash (high phosphate), 90 gms.; 300 ppm; bleach, 1 cup
- 5) Sears (carbonate), 90 gms.; 150 ppm; no bleach
- 6) Sears (carbonate), 90 gms.; 300 ppm; no bleach
- 7) Sears (carbonate), 90 gms.; 150 ppm; bleach, 1 cup
- 8) Sears (carbonate), 90 gms.; 300 ppm; bleach, 1 cup
- 9) Cold Water All (citrate), 1/3 cup; 150 ppm; no bleach
- 10) Cold Water All (citrate), 1/3 cup; 300 ppm; no bleach
- 11) Cold Water All (citrate), 1/3 cup; 150 ppm; bleach, 1 cup
- As the washer filled the water hardness concentrate was added to the water followed by the detergent. If used, bleach was added via the bleach dispenser. After water hardness and detergent had thoroughly mixed with the water, a wash load was added. This was done to avoid possible spot concentration of calcium carbonate build-up on the test fabric. After the wash cycle was completed, the wash load was tumble dried.

CONDITIONING OF SPECIMENS

Specimens were conditioned, burned and evaluated according to the specifications of DOC FF 3-71 which were possible. After test fabric units were laundered, 3 1/2" x 10" specimens were cut from them and excess fabric was reserved for calcium content determinations and photomicrographs. Ten specimens were taken from the 12" x 36" units and fifteen were taken from the 12" x 54" units.

Specimens were fixed between the plates of the specimen holders $(3\ 1/2" \times 16\ 5/8"$ with openings in the plates 2" x 14") which were held together by side clamps. Specimens were placed in a forced air circulating oven and maintained at 105 + 2.8°C (221 + 5°F) for thirty minutes.

After specimens were conditioned they were placed (in holders) in an air tight, moisture tight desiccating chamber using anhydrous silica gel as the desiccant. Not more than five specimens were placed in a desiccating chamber at a time. Specimens were allowed to cool in the desiccating chamber for at least thirty but not more than sixty minutes.

TEST CHAMBER

The test chamber used was a steel cabinet with inside dimensions of 29.8 cm. wide, 29.8 cm. deep and 75 cm. high. A 15.3 cm. diameter opening in the top of the test chamber provided ventilation. This was overlaid with a cover 20.4 cm. wide, 24.8 cm. deep and 3.2 cm. high that was open at the back. The cabinet was fitted with a closefitted door with a window 19.8 cm. wide and 40.3 cm. high for observation of the flame test. A support for the suspension of the specimen holders was placed parallel to the door of the cabinet halfway toward the back and 67.2 cm. up from the bottom. The burner was placed so that it could be moved below the specimen from one side of the cabinet. This cabinet differs insignificantly from the type specified for use in DOC FF 3-71 and was used on the basis of its availability.

The test chamber required by DOC FF 3-71 is slightly larger than the one used. It has a series of small ventilation ports in the top rather than one large one and a guide for the specimen holders. The support for the specimen holders is positioned so that the specimen plane is perpendicular to the

door of the cabinet rather than parallel to it as in the test chamber used.

SPECIMEN BURNING AND EVALUATION

The hood fan was turned off, and the flame was adjusted to a height of 3.8 cm. (1 1/2") above the highest point of the barrel of the burner. Specimens were removed from the desiccating chamber and suspended one at a time in the test chamber. The burner flame was impinged on the bottom edge of each specimen for 3.0 ± 0.2 seconds. The residual flame time was recorded to the nearest 0.1 seconds.

When the afterglow had ceased, the specimen was removed from the test chamber and the specimen holder and folded lengthwise along a line through the highest peak of the charred or burned area and creased by hand. The specimen was unfolded and a hook weighted with 113.4 gms. was inserted 1/4" from one corner of the lower edge. The other corner of the lower edge was raised slowly from the table. The char length (distance from the original lower edge of the specimen to the top of the tear or void) was recorded in centimeters. To pass the vertical flame test, the average char length of the five specimens in a sample could not exceed 17.8 cm. (7.0 inches). Also, no individual specimen could have a char length greater than 25.4 cm. (10.0 inches) or a residual flame time greater than 10.0 seconds.

CALCIUM CONTENT DETERMINATIONS

Calcium content was determined on test fabric units after 0, 16, 25 and 50 washings. Two determinations were made for each test fabric units. As a result of vertical flame test failures by the test fabric units laundered with the carbonate detergent, test fabric units within this group were

subjected to additional calcium content determinations after 4, 9, 36 and 55 washings. Analyses were by indirect EDTA back-titration as described by Wasserman and Basch (29). This method was chosen over that used by Pacheco and Carfagno as it required fewer reagents and procedure steps. The procedure was as follows. Fabric weighing 0.1 - 0.2 gms. was cut into small pieces and placed in an Erlenmeyer flask. The exact weight of the fabric was recorded. Ten milliliters of 0.01 M Na-EDTA solution, 15 - 20 milliliters distilled water and several drops of buffer (pH = 10) were added to the fabric and boiled for 10 - 15 minutes. The fabric was filtered through a sintered glass funnel and washed several times with hot distilled water. The solution was transferred to a 100 ml. Erlenmeyer flask for visual titration. The solution was brought to pH = 9.8 with a buffer (pH = 10). Several drops of indicator were added and the solution was titrated with 0.01 M CaCl₂ until the first color change occurred.

Calcium content (Ca + Mg) was calculated by the following formula and recorded as calcium percent of the fabric weight

$$\frac{(B - A) \times f \times 40}{w}$$
 = Calcium percent of fabric weight

A = ml. of 0.01 \underline{M} CaCl₂

B = m1. of 0.01 M Na-EDTA added to the fabric

f = factor of CaCl₂ standardized against Na-EDTA by titration
 used for determination

w = weight of fabric in milligrams

PHOTOMI CROGRAPHS

Exploratory scanning electron photomicrographs were taken to investigate

the microscopic appearance of selected test fabric samples as a result of various laundry conditions.

STATISTICAL ANALYSIS

An analysis of variance was conducted to determine if significant differences in residual flame time, char length and/or calcium content due to laundry variables existed. Data were analyzed by the AARDVARK program using a split-split-split plot design. To obtain equal classes for statistical analysis, ten specimens were randomly selected for consideration from test fabric swatches of fifteen specimens.

Chapter 4

RESULTS

Objective tests for this study were conducted in the laboratories of the Department of Clothing, Textiles and Interior Design at Kansas State University. Testing involved 600 wash cycles, 1095 vertical flame tests and 130 calcium content determinations.

VERTICAL FLAME TEST

According to DOC FF 3-71, two samples from each production unit must be tested for flammability after fifty washings. A production unit is rejected in the following instances:

- If a sample fails the 17.8 cm. average char length criterion.
- 2) If two or more individual specimens of the ten specimens from the two samples fail the 25.4 cm. char length and/or the 10 second residual flame time criteria.

Test fabric swatches rejected were:

Sears - no bleach - 150 ppm - 25 launderings

Sears - no bleach - 150 ppm - 36 launderings

Sears - no bleach - 150 ppm - 50 launderings

Sears - no bleach - 150 ppm - 55 launderings

Sears - no bleach - 300 ppm - 55 launderings

Sears - bleach - 150 ppm - 36 launderings

Sears - bleach - 150 ppm - 50 launderings

Sears - bleach - 150 ppm - 55 launderings

Sears - bleach - 300 ppm - 25 launderings

Sears - bleach - 300 ppm - 36 launderings

Sears - bleach - 300 ppm - 50 launderings

Sears - bleach - 300 ppm - 55 launderings

Residual Flame Time

The vertical flame time produced a go-no go situation. Most specimens burned less than one second. Some specimens burned for more than a few seconds but in such cases burned for more than 20 seconds. Samples containing such specimens, therefore, had higher average flame times.

When "Dash" detergent was used there seemed to be no meaningful change in flame time with increased number of washings or water hardness level, but the higher level of bleach appeared to cause a difference in flame time (See Figure 1, page 21).

With use of "Sears" detergent a dramatic increase in flame time was noted as a function of the number of washings. The level of water hardness and bleach appeared to cause no meaningful change in the residual flame time (see Figure 2, page 22).

With the use of "Cold Water All" detergent the number of washings and the water hardness level appeared to cause no meaningful change in flame time; however, the higher bleach level appeared to increase the flame time (see Figure 3, page 23).

Char Length

The burning of most specimens generated char lengths less than seven centimeters. A flame burning past seven centimeters usually consumed the entire length of the specimen (25.4 cm.).

THIS BOOK CONTAINS NUMEROUS PAGES WITH DIAGRAMS THAT ARE CROOKED COMPARED TO THE REST OF THE INFORMATION ON THE PAGE. THIS IS AS RECEIVED FROM

CUSTOMER.

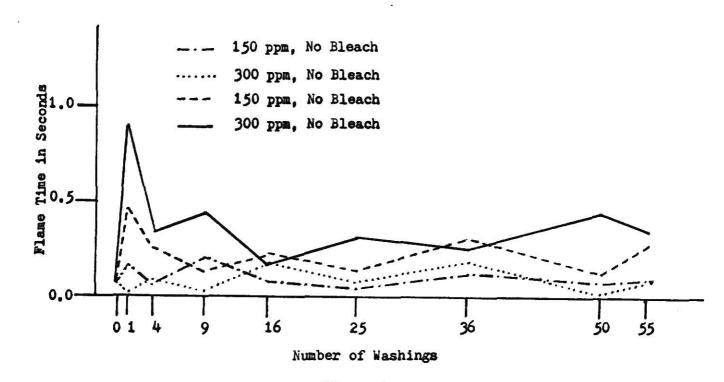


Figure 1

Average Residual Flame Times of Specimens Washed with "Dash" Detergent 0, 1, 4, 9, 16, 25, 36, 50 and 55 Times, with or without Bleach at 150 ppm or 300 ppm Water Hardness

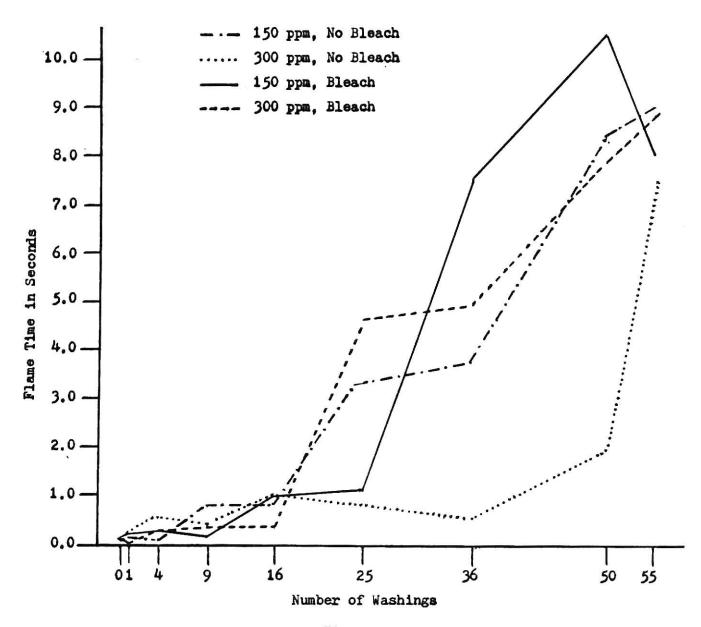


Figure 2

Average Residual Flame Times of Specimens Washed with "Sears" Detergent 0, 1, 4, 9, 16, 25, 36, 50 and 55 Times, with or without Bleach at 150 ppm or 300 ppm Water Hardness

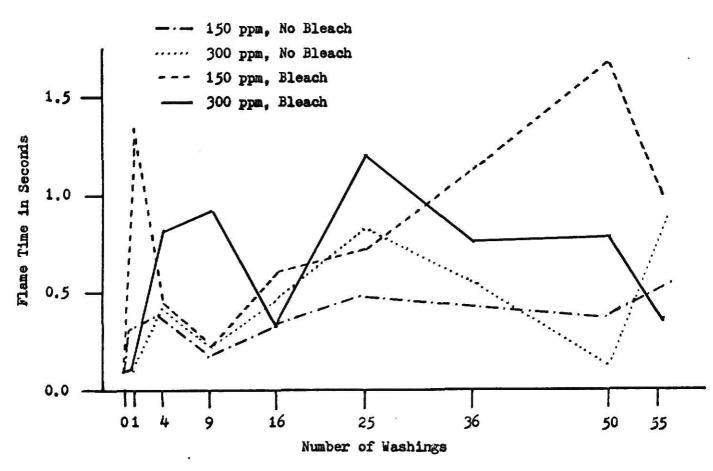


Figure 3

Average Residual Flame Times of Specimens Washed with "Cold Water All"
Detergent 0, 1, 4, 9, 16, 25, 36, 50 and 55 Times, with or without Bleach
at 150 ppm or 300 ppm Water Hardness

In fabric washed with "Dash" detergent it appeared that no meaningful changes in char length due to the variables existed (see Figure 4, page 25).

In fabric washed with "Sears" detergent the number of washings produced a meaningful change in the char length; the water hardness level and bleach condition did not (see Figure 5, page 26).

With "Cold Water All" detergent the char length increased with the use of bleach. No meaningful changes were noted due to the number of washings or the level of water hardness (see Figure 6, page 27).

CALCIUM CONTENT

Test Fabric washed with "Sears" detergent shows a definitely higher calcium content than fabric washed with the other two detergents (see Figures 7 and 8, page 28 and Figure 9, page 29).

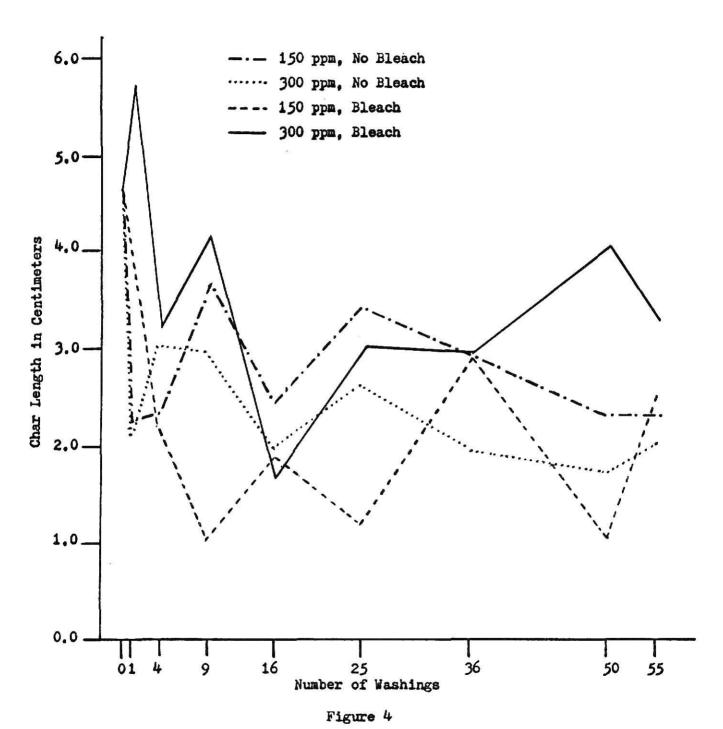
STATISTICAL ANALYSIS

Residual Flame Time

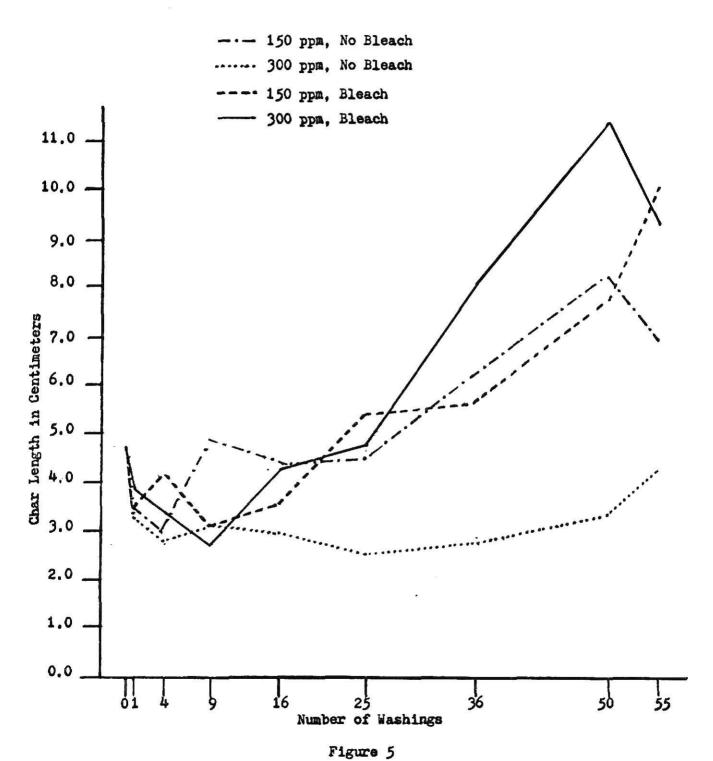
There were significant differences in flame times due to the detergent used. Specimens washed with "Sears" detergent had residual flame times much longer than those washed with "Dash" and "Cold Water All" detergents.

Detergent	Means(time in seconds)
Dash	0.171
Cold Water All	0.766
Sears	2.439
17 - 18 - 28 - 18 - 18 - 18 - 18 - 18 - 18	

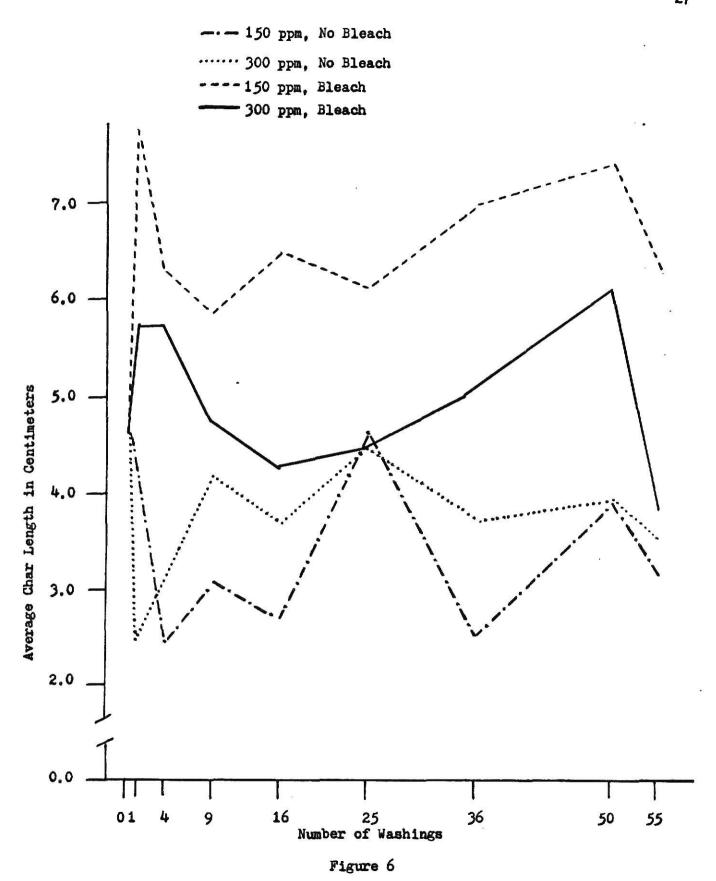
^{***}Significant at the .1% level



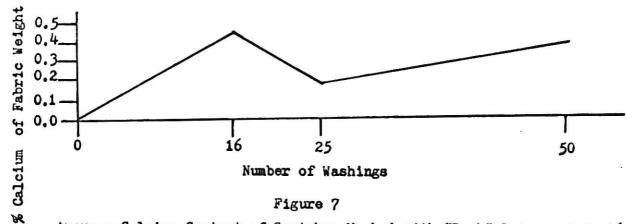
Average Char Lengths of Specimens Washed with "Dash" Detergent 0, 1, 4, 9, 16, 25, 36, 50 and 55 Times, with or without Bleach at 150 ppm or 300 ppm Water Hardness



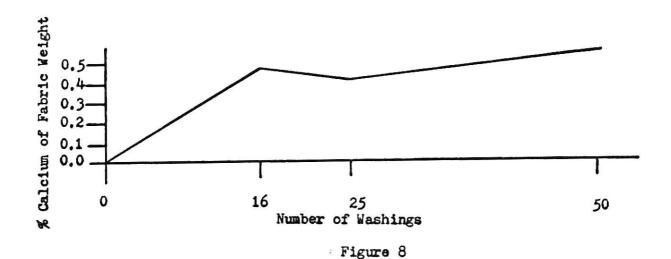
Average Char Lengths of Specimens Washed with "Sears" Detergent 0, 1, 4, 9, 16, 25, 36, 50 and 55 Times, with or without Bleach at 150 ppm or 300 ppm Water Hardness



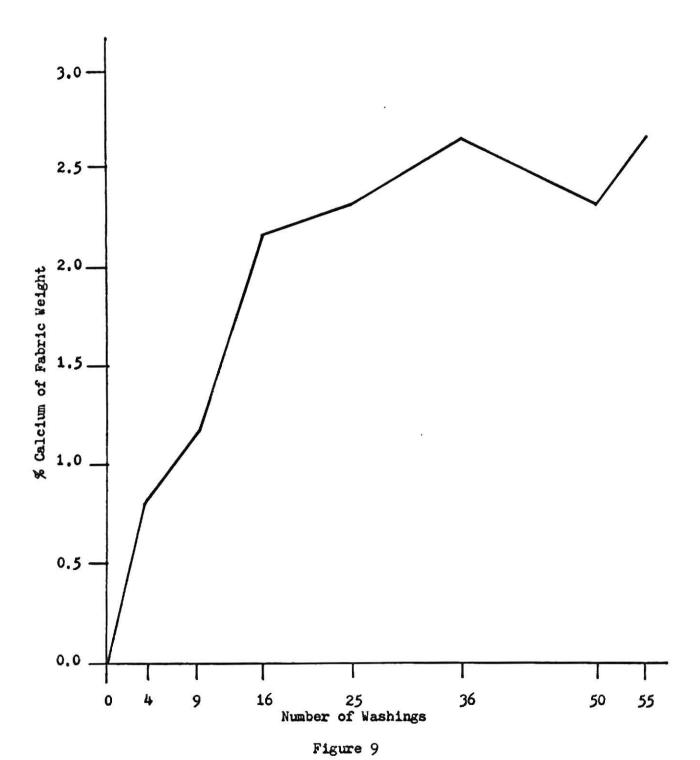
Average Char Lengths of Specimens Washed with "Cold Water All" Detergent 0, 1, 4, 9, 16, 25, 36, 50 and 55 Times, with or without Bleach at 150 ppm or 300 ppm Water Hardness



Average Calcium Content of Swatches Washed with "Dash" Detergent 0, 16, 25 and 50 Times, with and without Bleach at 150 ppm and 300 ppm Water Hardness



Average Calcium Content of Swatches Washed with "Cold Water All" Detergent o, 16, 25 and 50 Times, with and without Bleach at 150 ppm and 300 ppm Water Hardness



Average Calcium Content of Swatches Washed with "Sears" Detergent 0, 4, 9, 16, 25, 36, 50 and 55 Times, with and without Bleach at 150 ppm and 300 ppm Water Hardness

There were no significant differences in flame times due to bleach condition, water hardness level or the direction (warp or weft) in which the specimens were cut.

When the results for all conditions were combined some small significant differences were found due to the number of washings. With the exception of the zero level there was a trend.

Number of Washings	Means(time in seconds)
1	0.156
9	0.305
4	0.384
16	0.447
25	0.877
36	1.242 * 7
0	2.117 🗸 🚶
55	2.119
50	2.648

*Significant at the 5% level

No significant differences in flame time were noted due to interactions between controlled variables (i.e. bleach condition, water hardness level, etc.).

Char Length

A significant difference in char length was noted due to the detergent used. Fabric washed with "Dash" had shorter char lengths than did fabric washed with "Sears" and "Cold Water All" detergents.

Detergent	Means(in centimeters)
Dash	3.346 1
Sears	4.798 Ĵ
Cold Water All	4.896

^{*}Significant at the 5% level

Test fabric washed with bleach was shown to have much longer char lengths than that washed without bleach.

Bleach Condition	Means(in centimeters)
no bleach	3.533
bleach	5.136 Î

^{***}Significant at the 0.1% level

Test fabric washed in water with hardness of 150 ppm had much longer char lengths than that washed in water with hardness of 300 ppm.

Water Hardness	Means(in centimeters)
300 ppm	3.989
150 ppm	4.679

^{***}Significant at the 0.1% level

Calcium Content

In comparing the calcium content of test fabric significant differences in the amount of calcium were found due to the detergent used.

Means(in	8	Ca	of	fabri	ic weight
Dash			0.2	215]	
			0.3	337 Ĵ	1
\$\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\			1.7	705	Ĵ
				0.2	0.215 0.337 1.705

^{*}Significant at the 5% level

There was a significant difference in calcium content between original fabric and washed fabric.

Number of	Washings	Means(in % Ca of fabric weight)
0		0.000
16	3 50	0.976
25		1.015
50		1.018

^{***}Significant at the 0.1% level

For fabric washed with "Sears" detergent significant differences in calcium content due to the number of washings were found.

Number of	Washings	Means(in % Ca of fabric weight)
0	į	0.000
4		0.719
9	,	1.196
. 16		2.176
25	\$	2.282

^{***}Significant at the 0.1% level

Number	of Washings	Means(in % Ca of fabric	c weight)
	50	2.361	***
	55	2.561	
	36	2.684	٦

***Significant at the 0.1% level

PHOTOMI CROGRAPHS

Photomicrographs appear in Appendix B as Figures 2-10. Figure 2 shows fibers of test fabric before washing. Figures 3, 4 and 5 show fibers of test fabric washed 50 times with bleach and "Dash," "Sears" and "Cold Water All" detergents, respectively. Fibers in fabric before washing and washed with "Dash" and "Cold Water All" are relatively free of foreign material. Fibers of fabric washed with "Sears" shows heavy calcium carbonate deposits.

Figures 6 and 7 show fibers of test fabric washed in "Sears" and bleach at 300 ppm after 9 and 25 washings, respectively. Some deposition of calcium carbonate has occurred by the ninth washing. Heavy deposition has occurred by the twenty-fifth washing. Figure 8 shows fabric washed 50 times at 150 ppm with "Sears" and bleach. At the lower water hardness level fibers are heavily encased in calcium carbonate.

Figure 9 of fiber washed 50 times in "Sears" at 300 ppm without bleach reveals no fiber damage due to bleach by comparison. A higher magnification of calcium carbonate deposits is depicted by Figure 10.

Chapter 5

CONCLUSIONS

From the findings it was concluded that specific laundry variables can affect the flame retardancy of a Cordelan/cotton sleepwear knit.

In comparing the effects of laundry variables on the residual flame time, the type of detergent used proved to have the greatest effect. The Residual Flame time of fabric washed with the carbonate detergent was much greater than that washed with the phosphate and the citrate detergents.

The type of detergent was not responsible for great differences in char length. A longer char length was noted with the use of bleach and, oddly enough, the lower water hardness level. It is possible that fabric washed in water with 150 ppm water hardness had greater amounts of calcium remaining after 50 washings than did fabric washed in water with 300 ppm water hardness. It is believed that after the fiber was thoroughly coated with calcium carbonate no further deposition occurred. As evidenced from photomicrographs, it appeared that deposits eventually flaked off.

Fabric washed with the citrate detergent had a significantly greater calcium content than that washed with the phosphate detergent. Fabric washed with the carbonate had a very much greater calcium content than did that washed with the citrate detergent.

The number of washings had a great effect on the calcium content; however, after sixteen washings no significant differences in calcium content were noted. This finding indicates that the rate of deposition decreases as the number of washings increases. For the carbonate detergent alone great differences in calcium content were noted between 0, 4, 9 and 16 washings.

After sixteen washings differences became less and calcium content reached a peak at thirty-six washings and decreased thereafter.

Due to the go-no go nature of the vertical flame test, the validity of the findings of the statistical analysis in this situation is questionable. Due to the manner in which data were grouped for statistical analysis it was impossible to divide findings by detergent under each variable.

From these findings it was suggested that a carbonate detergent in combination with hard water be avoided in the laundry care of children's sleepwear made from a Cordelan/cotton fabric. Presumably, the use of a water softener would curtail the deposition when using a carbonate detergent.

It is doubtful that results could be precisely reproduced. An ASTM committee on textile materials carried out an interlaboratory study on the test methods of DOC FF 3-71; the sixteen laboratories involved failed to obtain the same pass-fail results in tests using the same fabrics. However, precision reproducibility is not necessarily expected from the flammability test, which is a go-no go test (21).

LITERATURE CITED

- 1. "Appearance of Durable Press Fabrics After Repeated Home Launderings, Method 124-1969," <u>AATCC Technical Manual</u>, 175-176.
- Baum, Burton M., "Flame Retardant Fabrics, Parts I, II and III," Chemtech, 1973.
- 3. Buck, George S., "The Case for Flammability," <u>Textile Industries</u>, 135 (2): 127-134•
- 4. "Can You Sell Flame Retardant Fabrics," Textile Industries . 135(2): 31-32.
- Chemical Qualities of Public Water Supplies in Kansas, Bulletin No. 1-7, Kansas State Department of Health, Environmental Health Service, June 1965.
- 6. Church, James M., "Flammability Hazards of Fabrics," Chemical and Engineering News, 31(1): 325-331.
- 7. Collins, J. R., "Flame Retardant Fabrics," <u>Plastics and Polymers</u> (Great Britain), 10:283-289.
- 8. "DOC FF 3-71 (as Amended) Standard for the Flammability of Children's Sleepwear," Textile Chemist and Colorist, 4(9):71-76.
- 9. "Durable Phosphorous-Containing Flame Retardants for Cellulose Textiles," Textile Chemist and Colorist, 4(2):43.
- 10. "Fiery Rebuttals at Flammability Conference," <u>Textile Industries</u>, 135(2): 123-126.
- 11. "Flame Resistant Fabrics Increasing," Modern Textiles, 55(11):44.
- 12. "Flame Retardants Directory," American Dyestuff Reporter, 61(1): 38-39, 40, 42, 44.
- 13. Gilliland, Barbara Faye, A Study of the Flame Retardant Properties and
 Thermal Behavior of Selected Flame Retardant Cottons (Master's Thesis),
 Cornell University, 1970.
- 14. Gilliland, B. F. and B. F. Smith, "Flame Retardant Properties and Thermal Behavior of Selected Flame Retardant Cottons," <u>Journal of Applied</u> Polymer Science, 16(7): 1801-1808.
- 15. Howry, Kenneth A., "New Approach to Testing," Modern Textiles, 55(12): 26-28.

- 16. Jancik, Helen Monju, Flammability of Selected Apparel Fabrics Treated with APO-THPC, THPC and THPOH-NH₃, (Master's Thesis), Louisiana State University, 1970.
- 17. Johnson, Annette J., A Comparison of the Flammability of Three Types of Fabric Used in Girl's Sleepwear (Master's Thesis), Oklahoma State University, 1968.
- 18. Kohjin Cordelan General Properties, Kohjin Co., Ltd., Osaka, Japan.
- 19. Kohjin Cordelan, Kohjin Co., Ltd., New York.
- 20. Le Blanc, Dr. Bruce, Textile Flammability Digest, 1(3):2.
- 21. Mandel, John, et al., "National Bureau of Standards Analysis of the ASYM Interlaboratory Study of DOC FF 3-71, The Standard for the Flammability of Children's Sleepwear," ASTM Standardization News, 1(5): 9-12.
- 22. Mazzeno, L. W., et al., "Degradation of Selected Flame Retardants on Exposure to UV and Elevated Temperatures," Textile Chemist and Colorist, 5(3):43.
- 23. Pacheco, J. F., and P. P. Carfagno, "How Laundering Practices Influence the Flame Retardancy of Fabrics," <u>Textile Chemist and Colorist</u>, 4(10): 255-259.
- 24. Perkins, R. M., G. L. Drake, W. A. Reeves, "The Effects of Laundering Variables on the Flame Retardancy of Cotton Fabrics," <u>Journal of the American Oil Chemist's Society</u>, 48(7):330-333.
- 25. Simms, D. L. "Fire Hazards of Fabrics," <u>Textile Institute and Industries</u>, 1(8):11-13.
- 26. St. John, Wayne L., Personal Communication, May, 1973.
- 27. Technology- Flame Retardant Vinyl Fiber," Chemical and Engineering News, 50(3):48.
- 28. Textile Fibers Technical Information Mutifiber, Bulletin X-45, DuPont Co., November 1955.
- 29. Wasserman, F. and A. Basch, "Determination of (Ca + Mg) in Flameproof Cotton Fabrics by EDTA Back Titration," <u>Textile Research Journal</u>, 43(2): 83-85.

APPENDIXES

APPENDIX A

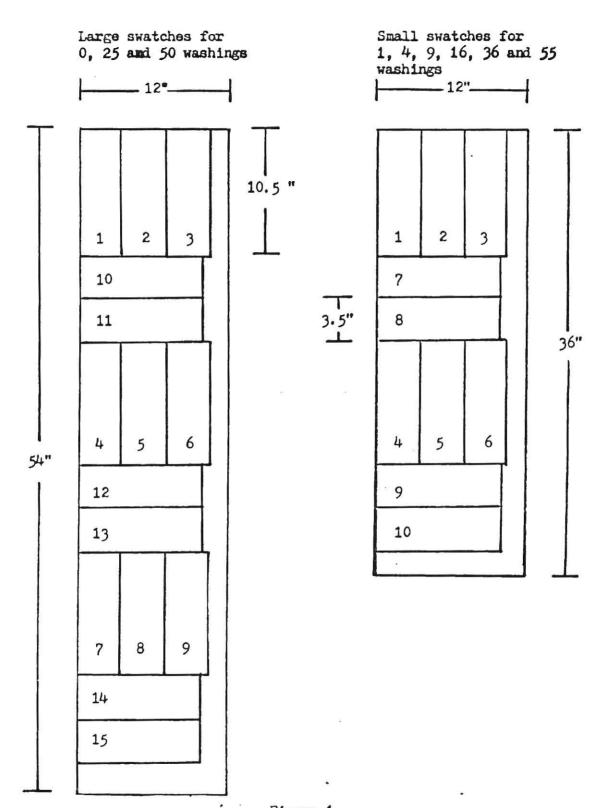


Figure 1
Cutting Plan for Test Fabric Specimens

APPENDIX B

SELECTED SCANNING ELECTRON PHOTOMICROGRAPHS OF FABRIC FROM THE STUDY

Each caption gives information on the washing conditions (detergent, bleach condition, water hardness and the number of washings) and magnification.

THIS BOOK CONTAINS SEVERAL DOCUMENTS THAT ARE OF POOR QUALITY DUE TO BEING A PHOTOCOPY OF A PHOTO.

THIS IS AS RECEIVED FROM CUSTOMER.

THIS BOOK
CONTAINS
NUMEROUS
PICTURES THAT
ARE ATTACHED
TO DOCUMENTS
CROOKED.

THIS IS AS
RECEIVED FROM
CUSTOMER.



Figure 2
Original Fabric, 400X



Figure 3

Dash Detergent, Bleach, 300 ppm, 50 Washings, 400X



Figure 4
Sears Detergent, Bleach, 300 ppm, 50 Washings 800X

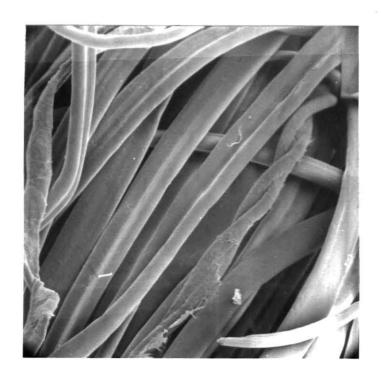


Figure 5

Cold Water All Detergent, Bleach, 300 ppm, 50 Washings, 400X

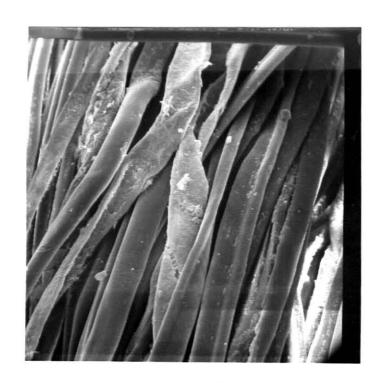


Figure 6
Sears Detergent, Bleach, 300 ppm, 9 Wash-ings, 400X

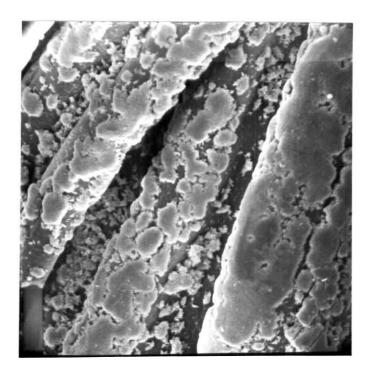


Figure 7
Sears Detergent, Bleach, 300 ppm, 25 Washings, 1600X



Figure 8 Sears Detergent, Bleach, 150 ppm, 50 Washings, 400X



Figure 9
Sears Detergent, No Bleach, 300 ppm, 50 Washings, 400X

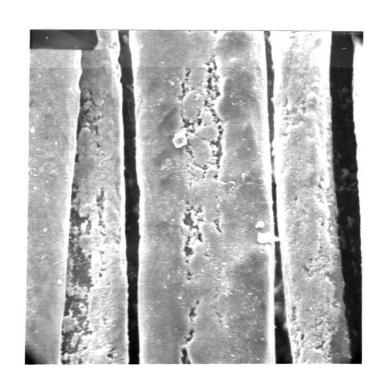


Figure 10
Sears Detergent, No Bleach, 300 ppm, 50 Wash-ings, 1600X

THE EFFECTS OF LAUNDRY VARIABLES ON THE FLAME RETARDANCY OF A CORDELAN/COTTON SLEEPWEAR KNIT

by

SELMA SUE FENT

B. S., Kansas State University, 1970

AN ABSTRACT OF A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Clothing, Textiles and Interior Design

KANSAS STATE UNIVERSITY Manhattan, Kansas

1973

The effects of laundry variables on the flame retardancy of a 87 1/2% Cordelan/12 1/2% cotton sleepwear knit fabric were studied. Laundry variables included detergent type (high phosphate, carbonate and citrate), water hardness (150ppm and 300ppm), bleach condition (bleach and no bleach) and the number of washings (0, 1, 4, 9, 16, 25, 36, 50, and 55). Fabric specimens were subjected to a vertical flame test and evaluated as specified by the Standard for the Flammability of Children's Sleepwear, DOC FF 3-71. Calcium content was determined by EDTA back titration. Photomicrographs were taken of selected specimens.

Residual flame times were significantly lower for fabric washed with carbonate detergent than for fabric washed with the phosphate or citrate detergents. The residual flame time increased with the number of washings.

Char lengths were significantly longer for fabric washed with carbonate and citrate detergents than for fabric washed with phosphate detergent. Fabric washed with bleach produced longer char lengths than did those washed without bleach. Longer char lengths resulted when fabric was washed in water with 150 ppm hardness rather than 300 ppm hardness.

Calcium content was significantly higher in fabric washed with the carbonate detergent. Calcium content increased with the number of washings.