

THE INCORPORATION OF A CHELATING AGENT INTO A FLAME  
RETARDANT FINISH ON A COTTON FLANNELETTE AND THE  
EVALUATION OF SELECTED FABRIC PROPERTIES

by

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## CHAPTER I

### INTRODUCTION

When the Standard for the Flammability of Children's Sleepwear, DOC FF 3-71 (1), became effective, there was considerable resistance to it by many industrial spokesmen. This resistance was aimed primarily at the testing method and the expense of compliance. These objections, however, lessened due to the many chemical treatments available (2)(3)(4) and the fact that the industry did gear itself for the necessary testing involved. The emphasis has continued on fabric properties, such as increased durability, improved hand, prevention of odors, and fabric care.

One of the standard's requirements was that the flame resistant garment must pass the burning test after fifty launderings. In testing the treated fabrics to determine this durability, it was discovered that the use of carbonate based non-phosphate detergents or soaps could impair the fire resistant properties of the fabric (5)(6)(7)(8). Laboratory testing showed that soap, carbonate, and silicate products in solution with hard water accumulate as deposits on the fabric (9). The use of soap in soft water was not found to alter the flame resistance (10)(5).

The cause for the decrease in resistance to flaming was determined to be an ion exchange between the fabric and the metals in the hard water (11). The metal ions, usually calcium or magnesium, were found to be attracted to the fiber surface by the negatively charged cellulose which resulted in bonding between the carboxy end groups and the metals (11).

Reeves (12) determined that phosphorous, which is used in most household and apparel flame retardant finishes (5), contributed to flame resistance by producing acid groups which esterified cellulose and subsequently promoted dehydration during the pyrolysis of the treated cotton. It was also reasoned that since the calcium and magnesium salts were thermally stable, they were incapable of esterifying cellulose and therefore, did not contribute to the flame resistance (12).

Although care labeling on flame retardant garments usually states that washing should be done with a detergent, a recent survey of consumer laundry practices found that consumers did not differentiate between the terms "soap" and "detergent" and that they used the two laundry products interchangeably (13).

Most detergents for home and commercial laundering contain phosphates. The negative phosphate ions attract the positively charged calcium or magnesium ions in the water thereby preventing the formation of deposits on the fabric. However, due to the polluting effect of nonbiodegradable phosphates, the consumer is asked to reduce or eliminate their

use; in some states, phosphates are completely banned from use (9). Although an acid sour of acetic acid would remove any deposits from the fabric, this is an unacceptable alternative since, after approximately ten treatments, the finish would probably be destroyed by acid hydrolysis (5).

The use of a chelating agent to remove metal ions from water, that is to make "soft" water, has been extensively researched (14). The theory of chelation has, basically, stated that one or more rings form when a metal ion combines with an electron donor which has two or more flexible donor groups capable of forming stable ring structures. The sequestering agent was shown to inactivate the metal ions in solution by combination with them. Two examples of organic chelating agents were listed as ethylenediamine tetraacetic acid (EDTA)(14) and carboxymethyl cellulose (CMC).

The characteristics of a chelating agent would reduce the need for high phosphate detergents if they could be incorporated into flame retardant finishes or fabrics since most of the metallic ions would be prevented from forming solids on the fabric.

As the water in most areas of the United States is hard (12), the consumer must decide between retaining the non-flammable finish effects on their children's sleepware and continued pollution of water. By eliminating the need for phosphates in detergents, the pollution conflict would be partially solved.

The foregoing factors led to this researcher's interest in determining the effect of a chelating agent as an integral part of the flame retardant finish.

The objectives of the research are as follows:

1. To determine the ability of including a chelating agent in a flame retardant finish without altering the non-flammable properties.
2. To determine the effect of a chelating agent on flame retardant fabrics washed in hard water with soap.

## CHAPTER II

### REVIEW OF LITERATURE

With the advent of government standards concerning children's sleepwear, the manufacturers of cotton flannelette have become very interested in procuring a finish which is economical and yet passes the requirements specified in DOC FF 3-71 and the soon to be enacted DOC FF 5-74 (15).

The interest in flame-resistant flannelette did not begin with proceedings resulting in the enactment of DOC FF 3-71, but was a cause of concern in the early 1900's. William Henry Perkin became interested in flannelette's extreme flammability around 1913 and, as a result, conducted a series of researches which led to the development of a commercial process called "Non-Flam" (11). This process called for the impregnation of the cotton flannelette with sodium stannate and then ammonium sulfate followed by washing and drying (16). Stannic oxide was believed to be the retardant which in some way reacted with the cellulose and became resistant to laundering with soap. The process did not win much interest, however, and little more was done on flameproofing until after World War II (11).

The mechanism by which a fabric burns is a complicated phenomenon. A flammable material will ignite if a flame

attacks it when sufficient oxygen is available. The heat which is produced during the burning causes a transfer of the surrounding atmosphere and/or penetrates into the solid. As the surface regresses, solid particles are drawn into the flame thereby acting as fuel. This indicates that the rate of surface regression is a function of available oxygen and the rate at which particles are fed into the flame. That heat which diffuses into the solid causes pyrolysis, an endothermic chemical degradation process (17). The pyrolysis results in a solid char, a liquid distillate, and a gaseous fraction (16). The latter two products are highly volatile and diffuse to the surface of the solid to form additional fuel and thereby increase the flame's temperature.

Radical chain reactions which cause flaming combustion are formed by the pyrolysis of the fabric. Afterglow is merely an oxidation of char by air to give off carbon dioxide and some carbon monoxide (17).

The mechanism of flame retardancy is complicated both due to the complexities of burning itself and due to the wide range of compounds used as flame retardants. Little (16) divides the mechanism into four broad areas: chemical, coating, gas, and thermal theories.

The chemical theories deal with those compounds which have a pronounced degrading action on cellulose with the presence of heat. These compounds alter the normal course of degradation and instead form lesser amounts of inflammable

tars and gases thereby increasing the production of non-volatile carbonaceous material.

It was known as early as 1820 that certain flame-retarding salts have low melting points and would coat the fabric with a slick layer upon contact with heat. The coating theory basically involves the covering of cellulose with an impermeable skin thus excluding oxygen and preventing the continuation of the combustion reaction (16)(17). The action of borax and boric acid has been explained with this theory since this mixture leaves a glassy matrix which forms a tightly adhering cover over the cotton fibers.

Gas theories assume that by treating fabrics with materials that easily decompose at elevated temperatures and produce inert or non-oxidizable gases or vapors, the atmosphere in the area of the fabric will change. This change supposedly either dilutes the flammable gases produced by normal cellulosic decomposition or excludes the normal atmosphere from the area; that is, gives a blanketing effect. Although this theory has been generally accepted, experimentation (18) has shown that the quantities of inert gases produced are not much greater than the fabric alone. This theory has been thought to be more of a partial explanation of flame retarding mechanisms rather than a theory of major importance.

Thermal theories contain two major concepts. The first of these assumes that the amount of heat from a source may be

scattered by an endothermic change in the flame retardant itself; thus any phase transitions made by the retardant mixture might absorb a large amount of energy which would otherwise contribute to fabric combustion. The second concept assumes that the heat may be conducted away from the fibers at the same, or a similar, rate at which the source supplies it, thereby preventing the fabric at the edge of the flame from ever reaching its combustion temperature (16).

Flame retardancy can be achieved by affecting any or all of the stages of burning polymers and the retardant may incorporate more than one of the theories stated above in preventing flame propagation. Phosphorous containing flame retardants react primarily in the solid phase and therefore affect the degradation of the polymer. Halogens, however, act predominately in the gas phase but may also act in the solid phase (17).

The amount and chemical nature of the flame retardant has the greatest effect on the burning characteristics of cotton. Atmospheric conditions, mainly humidity and air movement, and the physical and chemical properties of the fabric, such as weight, weave, yarn size, type of dye, and type of finishing agent, also affect the way a flame retardant cotton fabric burns. The most important class of durable or wash fast flame retardants are the phosphorous containing materials which usually contain either nitrogen or bromine or sometimes both (19).

Nitrogen, a constituent of flame retardants for many years, was initially thought to permit the salts, ammonium phosphate and ammonium sulfamate, to decompose at lower temperatures than for the corresponding metal salts. Later, the nitrogen was recognized for its buffering abilities since it prevented excess degradation of the cellulose. It also reduced the ion exchange properties of the phosphorylated cotton thereby minimizing the effects of hard water during laundering (19).

The incorporation of nitrogen into a phosphorous containing flame retardant has been found to increase the flame retarding efficiency (17)(11)(16) thereby exhibiting a synergistic effect. The study of synergisms is a relatively new field of investigation and therefore only a limited number of systems has been studied (17). For the phosphorous-nitrogen synergism, Hendrix (20) has shown that, in the presence of urea, some organic phosphate forms phosphoramidate which acts as a flame retardant for cotton.

The most widely used flame retardants are based upon tetrakis (hydroxymethyl) phosphonium chloride (THPC) (21). THPC is produced in high yield from the reaction of formaldehyde with phosphine and hydrochloric acid (22). Four methylol phosphorous derivatives which are flame retardants include THPC-amide, THPOH-NH<sub>3</sub>, THPOH-TMM-NH<sub>3</sub>, and THPOH-amide.

THPC-amide is applied to cotton by a pad-dry-cure process (23). The finish is durable to home and commercial

laundering and imparts rot resistance but leaves the fabric with a stiff hand and a reduction in tensile and tear strength. Modifications to this flame retardant include the use of antimony oxide and organic chlorine compounds as a substitution for part of the THPC-amide, allyl phosphazine- $\text{CHBr}_3$  which enables a reduction of the combined formulation, allyl phosphate- $\text{CHBr}_3$  which only coats the fibers instead of penetrating them as THPC-amide does, and tris(2,3-dibromopropyl) phosphate which is used for treating polyester/cotton blends (21). The main object of these modifications is to reduce the cost and to improve fabric qualities.

THPC can be converted to tetrakis (hydroxymethyl) phosphonium hydroxide (THPOH) by reacting 1 mole of THPC with .8 to .9 mole of sodium hydroxide to a pH of about 7 or 7.5 (2). The retardant is applied by padding the fabric with a solution of THPOH, partially drying the fabric, and then exposing it to ammonia gas. It is important not to completely dry the fabric before exposure to the ammonia since the THPOH will decompose to less reactive compounds (2).

The THPOH- $\text{NH}_3$  flame retardant does not stiffen the fabric and results in little, if any, strength loss. If fabric softeners are not used, the strength is usually greater than the untreated fabric (21).

THPOH-TMM- $\text{NH}_3$  employs the use of trimethylolmelamine (TMM) plus wetting and softening agents as desired. The finish is padded onto the fabric, partially dried, exposed to ammonia

and then heated at 150°C for three minutes. TMM is substituted for approximately one-third of the THPOH required in the THPOH-NH<sub>3</sub> process. Properties imparted to the fabric by this retardant are the same as for THPOH-NH<sub>3</sub> except that breaking strengths do not increase and the cost of the chemicals is less; when THPOH alone is used, the cost is about 12 cents per square yard while with TMM added in a 2:1 ratio of THPOH to TMM, the cost drops to about 8.7 cents per square yard (21).

The THPOH-amide flame retardant, the fourth of the methylol phosphorous derivatives, is applied in a manner very similar to that of THPOH-TMM-NH<sub>3</sub> (24). The fabrics retain approximately 80-90% of their original breaking strength, are less stiff, have a high degree of rot resistance, and have a reduced tendency toward yellowing after exposure to sodium hypochlorite.

N-methyl dialkyl phosphonopropionamide is another type of flame retardant which is commercially available (25). When the fabric is treated with a 30-35% solids add-on, the result is nearly undistinguishable from untreated cotton in stiffness (16). The finish is a liquid, miscible with water in all proportions, non-toxic, and requires no ammonia cure. Conventional finishing equipment can be used and the retardant is fast to repeated launderings and dry cleanings. When exposed to heat, N-methyl dialkyl phosphonopropionamide induces relatively low temperature carbonization of the fabric thereby suppressing the release of flame-propagating tars.

Many other types of flame retardant finishes are available commercially but are not covered in this review. The researcher does not wish to recommend any of the above mentioned flame retardants as being more advantageous but only wishes to discuss those finishes most frequently cited in the relevant literature.

The effects of hard water and soap in laundering flame retardant sleepwear has proven to be detrimental to the finish (26)(27), and so phosphate detergents are recommended. These phosphates can contribute to the ageing of lakes by a process called eutrophication in which the lakes become choked with vegetation and sediment. With an acceleration of eutrophication, large masses of floating algae can result. There is controversy over how much damage the phosphorous does but consumers are urged not to use phosphate detergents until these differences are resolved (28).

The use of a chelating agent to make water soft is very common (14). Condensed phosphates are the most common of the sequestering agents since they are manufactured in large quantities. Those polyphosphates, in addition to inactivating metal ions, can deflocculate and suspend water-insoluble substances, thus offering a special advantage to laundering. They also prevent a precipitate formation in hard water when such conditions are made favorable by the presence of very small amounts (under 20 ppm) of polyphosphate (14). A disadvantage of the polyphosphates is their tendency to hydrolyze to the

orthophosphates in aqueous solutions, especially at elevated temperatures.

Organic chelating agents are becoming more popular as their price decreases. Ethylenediamine tetraacetic acid (EDTA) a typical commercial sequestering agent, is thermally stable in water and does not decompose at elevated temperatures. The stabilities of the chelates formed are much higher than those of polyphosphates which results in a lowering of metal-ion concentration.

Chelating agents are used in detergents to aid in the removal of metal salts from wash water. Nitrilotriacetic acid (NTA) has been used as a replacement for phosphates but research which pointed toward the possibility of its causing cancer in rats has discouraged further use (29). CMC is also used in detergents although its main purpose is for the anti-deposition of soil on clothing while it is being laundered (30)(31). No use of CMC purely as a chelating agent with the intent of removing the metal ions in hard water and preventing the precipitation of stearates and oleates on fabric was found by this researcher.

## CHAPTER III

### PROCEDURE

This research evaluated the use of a chelating agent as part of the flame retardant finish to determine its effects upon flammability after being laundered under specific conditions.

#### Description of Test Fabric

The evaluation was carried out using a 4.19 oz. per square yard, 100% blue cotton flannelette fabric with a construction of 49X49 yarns per square inch. Samples were cut 11"X12" so that three burning samples could be obtained from one large sample. All burning samples were cut only in the warp direction.

A commercially treated flame retardant fabric was used as a control to ensure the durability of the finish applied in this research. The fabric was 5.39 oz. per square yard with a construction of 47X45 yarns per square inch. This fabric was also 100% cotton flannelette.

#### Description of Pad Baths

A control bath without CMC and an experimental bath with CMC were made up as follows:

BATH I    35% N-methyl dialkyl phosphonopropionamide  
           10% triazine-formaldehyde resin  
           2% nonionic polyethylene emulsion--softener  
           4% buffered organic amine salt--catalyst  
           49% distilled water

BATH II    35% N-methyl dialkyl phosphonopropionamide  
           10% triazine-formaldehyde resin  
           2% nonionic polyethylene emulsion--softener  
           4% buffered organic amine salt--catalyst  
           46.5% distilled water  
           2.5% CMC

#### Finish Application Procedure

1. Prior to sample cutting, the fabric was scoured in a Najort<sup>R</sup> reversible wash-wheel machine at 100°F for 25 minutes with 90 grams of AATCC\* Test Detergent 124. The fabric was thoroughly rinsed, spun in a centrifugal extractor and dried in an electric tumble dryer.
2. Test samples were cut 11"X12" according to the directions under Description of Test Fabric.
3. The pad bath solutions were made according to the percentages listed under Description of Pad Baths.
4. The test samples were allowed to soak individually for ten minutes in the pad baths and then the excess finish was removed by squeezing the fabric twice through a wringer to achieve a wet pickup of 129.9% on Bath I samples and 197.5% on Bath II samples.

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Najort<sup>R</sup>--Trademark for Robert Ewing and Sons, Inc.

\* American Association of Textile Chemists and Colorists

5. All samples were allowed to air dry for a period of 12 hours.
6. Each sample was cured individually by placing on a pin frame and into a gravity-fed oven at a temperature of 171°C for three minutes.
7. An afterwash of 2% sodium carbonate at 140°F was then applied to the samples and was followed by a final rinsing in tap water.
8. The samples were placed in an electric tumble dryer for 30 minutes.
9. All samples were then overcast by sewing machine to prevent raveling during laundering.

#### Laundry Procedure

1. Bath I samples were laundered separately from Bath II samples. Calcium chloride (5.5 grams) was added manually to each wash and rinse cycle to bring the hardness of the water to approximately 140 ppm calculated as  $\text{CaCO}_3$ . The hardness was confirmed on the first, third, fifth, and ninth wash and rinse cycles by a standard soap solution titration (32). For each wash cycle, 90 grams of soap was added.
2. All laundering was done in a Sears Kenmore 70 Model 20670 Series washing machine. Terry cloth towels were used to maintain a four pound load, and drying was done in a Maytag electric tumble dryer. The samples were washed and

dried in sequence as would be expected of home laundry procedures.

3. Laundering procedures followed AATCC 124-1973 (33) as specified in DOC FF 3-71. Launderings were done through five and ten cycles rather than fifty. The wash load was removed from the machine during all spin cycles and, instead, spun in a centrifugal extractor for two minutes after the wash cycle and five minutes after the rinse cycle. Water temperature was checked on the first, third, fifth, and ninth washes.
4. Marked samples were removed after five and ten wash/dry cycles.

#### Test Procedures

1. The flammability tests were carried out as specified in DOC FF 3-71 except that only three samples in the warp direction were used rather than five samples in both warp and filling directions. Based on the fabric weight as determined by the test procedure, a .25 lb. weight was used to determine the char length. The times of the burns (seconds) and the char lengths (inches) were recorded on all samples.
2. Test samples were examined under the scanning electron microscope at magnifications of 350X and 1500X. Samples treated with Bath I and Bath II were examined before washing and after five washes. A sample treated with

Bath II was examined also after ten washes.

## CHAPTER IV

### RESULTS AND DISCUSSION

Data for the following was obtained according to the procedures described in Chapter III. Char lengths and burn times were the values recorded. The following paragraphs describe observations recorded during testing but which could not be statistically analyzed.

#### Uniformity of Finish Application

It was found very difficult to treat the test fabric with the 2.5% CMC solution (Bath II) due to its high viscosity. The test samples were allowed to soak for ten minutes to give the cellulose time to swell and permeate the CMC within the crosslinking resin. The samples were squeezed through a wringer twice to assure uniformity of application.

#### Curing Time and Temperature

To ensure an adequate and efficient cure time and temperature, Bath I samples, Bath II samples, and the commercially treated fabric were washed 25 times in soft water (approximately 50 ppm as  $\text{CaCO}_3$ ) with 90 grams of AATCC Test Detergent 124. Average char lengths and burn times were recorded (Table 1). Burning was carried out according to the procedure

Table 1  
AVERAGE CHAR LENGTHS AND BURN TIMES OF SAMPLES  
TESTED FOR DURABILITY OF FINISH APPLICATION

Sample	Char length/inches	Burn time/seconds
Commercially treated fabric	1.29	1.05
Bath I Samples	1.13	1.02
Bath II Samples	1.32	1.71

outlined in DOC FF 3-71 except that the samples were conditioned at a relative humidity of  $65 \pm 2\%$ . The samples were then mounted and placed in plastic bags to maintain conditioning and tested within 10 seconds of removal from the plastic bag.

Since the laboratory prepared samples compared to the commercially treated fabric in finish durability, no more work was done on the curing time and temperature for the finish.

#### Fabric Hand and Odor

The hand of the CMC treated samples was stiff after curing as compared to the untreated cotton; after one wash, the fabric hand improved and continued to soften with subsequent launderings. Those samples treated in Bath I retained a hand very similar to the untreated cotton.

All the laboratory finished samples had a distinct odor after curing which diminished as laundering progressed.

#### Method of Laundering

In loading the machine, all test samples were put in first and allowed time to wet out. The dummy load of terry towels was then added and after becoming wet the soap was added. This sequence was followed in all launderings to give the CMC time to react with the metal ions in the water before the soap deposits could form.

The wash load was removed from the washing machine and spun in a centrifugal extractor due to the inability of the researcher to control the water hardness during the spray action of the spin cycle. Spin times in the extractor were similar to those of the washing machine, however, the speed of spinning was probably faster.

### Water Hardness

The water used in this research was determined to have an initial hardness of approximately 50 ppm as  $\text{CaCO}_3^*$ . It was calculated that with the volume of water held by the washing machine, 5.5 grams of  $\text{CaCl}_2$  would increase the water hardness by 90 ppm as  $\text{CaCO}_3$  and thus result in a total hardness of approximately 140 ppm.

### Fabric Appearance

Both Bath I samples and those treated with Bath II displayed a light streaking after five launderings and a more pronounced color change after ten launderings. This loss of color was evident in the warp direction only and was no more than one-half inch in width. These areas were avoided as much as possible when cutting samples for burning.

### Effects of CMC Concentrations

When experimental bath samples containing 1.5%, 2.0%,

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\*As reported by the Blacksburg-Christiansburg & VPI Water Authority

and 2.5% CMC were washed together with Bath I samples in 150 ppm hard water with 90 grams of soap, a protective effect seemed to occur on the Bath I samples. Average burn times and char lengths were recorded and are shown in Table 2.

#### Interpretation of Data

For both the Bath I samples and Bath II samples, twelve replications of char length and time of burning were recorded. Tables 3, 4, 5, and 6 contain the char lengths and burn times recorded for the treated samples.

After five launderings with soap in hard water, the Bath I samples all burned the entire length of the sample, 10 inches, with an average burning time of 16.18 seconds. Those samples treated with Bath II had an average char length of 4.35 inches and an average burn time of 5.23 seconds.

Although Bath II sample No. 7 did burn the entire 10 inches, it was felt that nonuniformity of treatment caused the increased burning. The remaining Bath II samples all showed an improvement over the Bath I samples in both char lengths and burn times; these samples also passed the criterion set by the governmental standard DOC FF 3-71 which states that the average char length should not exceed 7.0 inches.

Bath I samples laundered ten times in the hard water with soap all burned the entire ten inches and the average burn time increased by .385 seconds. Samples treated with

Table 2  
AVERAGE CHAR LENGTHS AND BURN TIMES FOR DIFFERING  
CONCENTRATIONS OF CMC ADDED TO THE  
FLAME RETARDANT TREATMENT  
FIVE LAUNDERINGS

Sample	Char length/inches	Burn times/seconds
Bath I	5.13	4.78
1.5% CMC	5.25	5.78
2.0% CMC	5.33	5.41
2.5% CMC	7.92	6.79

Table 3  
CHAR LENGTHS AND BURN TIMES FOR BATH I SAMPLES  
FIVE LAUNDERINGS

Sample No.	Char length/inches	Burn time/seconds
1	10.00	13.7
2	10.00	23.0
3	10.00	15.7
4	10.00	25.5
5	10.00	15.8
6	10.00	14.8
7	10.00	14.0
8	10.00	29.4
9	10.00	9.7
10	10.00	14.0
11	10.00	12.3
12	10.00	12.3

Table 4  
CHAR LENGTHS AND BURN TIMES FOR BATH II SAMPLES  
FIVE LAUNDERINGS

Sample No.	Char length/inches	Burn time/seconds
1	4.62	6.2
2	5.84	5.4
3	4.72	5.0
4	4.30	4.4
5	3.80	5.0
6	2.54	3.3
7	10.00	16.0
8	2.32	2.5
9	2.64	1.6
10	3.74	3.9
11	2.54	2.5
12	5.24	7.1

Table 5  
CHAR LENGTHS AND BURN TIMES FOR BATH I SAMPLES  
TEN LAUNDERINGS

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Sample No.	Char length/inches	Burn time/seconds
1	10.00	27.2
2	10.00	16.8
3	10.00	12.9
4	10.00	14.9
5	10.00	17.1
6	10.00	13.0
7	10.00	10.8
8	10.00	13.5
9	10.00	25.5
10	10.00	14.2
11	10.00	27.4
12	10.00	11.5

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Table 6  
CHAR LENGTHS AND BURN TIMES FOR BATH II SAMPLES  
TEN LAUNDERINGS

Sample No.	Char length/inches	Burn time/seconds
1	10.00	18.2
2	10.00	8.8
3	10.00	14.5
4	10.00	14.7
5	10.00	17.1
6	10.00	13.9
7	10.00	10.6
8	6.73	5.8
9	8.88	7.3
10	10.00	17.7
11	10.00	18.3
12	10.00	9.9

Bath II which were laundered ten times showed a marked increase in both char length and burn time over the samples washed five times. All but two of the Bath II samples burned the entire length which resulted in an average char length of 9.63 inches and an average burn time of 13.07 seconds.

Nonuniformity of application or of soap deposition may have contributed to the extinguishing of the two Bath II samples laundered ten times.

#### Photomicrograph Analysis

Examination under the scanning electron microscope of the fabric samples before and after laundering showed the deposition of soap curd that coated the outside edges of the fibers. After application and curing of Bath I, the cotton fibers were shown as being free from any debris or deposits (Figures 1 and 2). After five launderings, however, (Figures 3 and 4) these deposits could be seen which probably caused the fabric to burn the entire length of the test strip.

Bath II samples, before laundering (Figures 5 and 6), showed a deposit which was concluded to be the CMC particles adhering to the fiber surface. After five launderings (Figures 7 and 8), a build-up of what was believed to be calcium salt deposits was shown as beginning to encase the fibers.

Upon completion of ten launderings of the Bath II samples, the calcium salt deposits seemed to have been

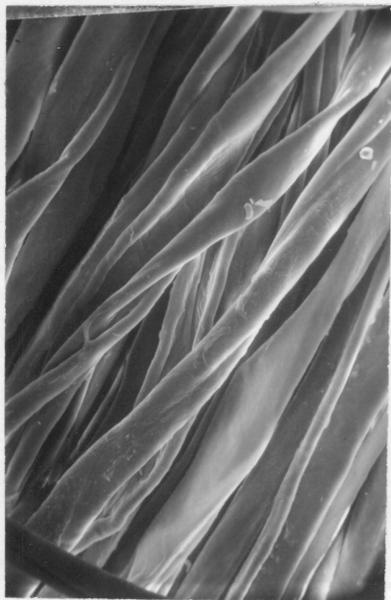


Fig. 1.--Bath I sample  
before laundering  
(375X)

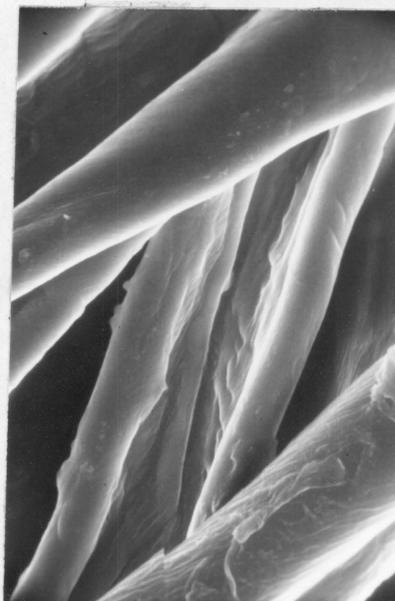


Fig. 2.--Bath I sample  
before laundering  
(1500X)

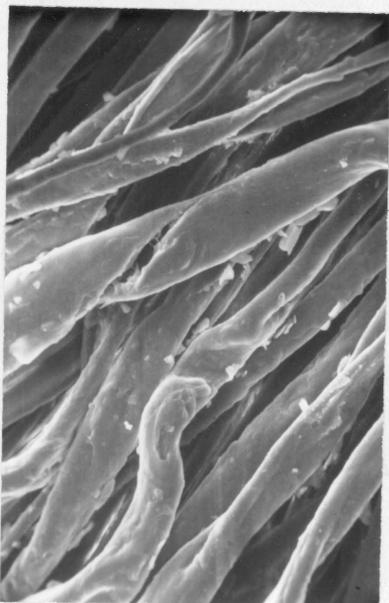


Fig. 3.--Bath I sample  
laundered 5 times in  
hard water with soap  
(375X)

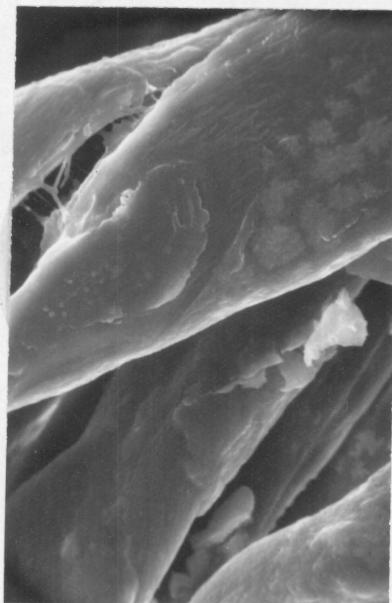


Fig. 4.--Bath I sample  
laundered 5 times in  
hard water with soap  
(1500X)

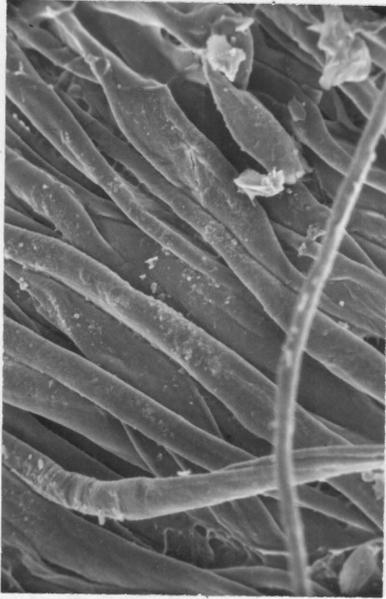


Fig. 5.--Bath II sample  
before laundering  
(375X)

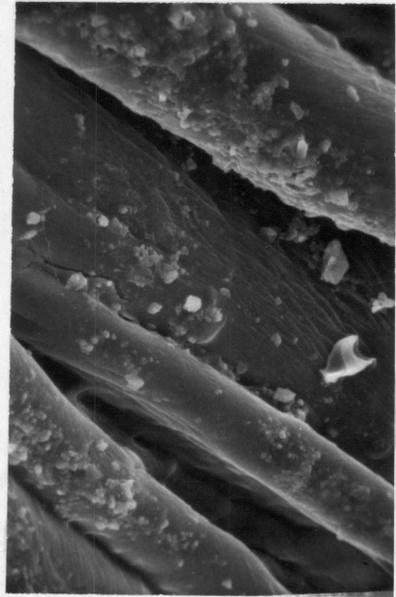


Fig. 6.--Bath II sample  
before laundering  
(1500X)

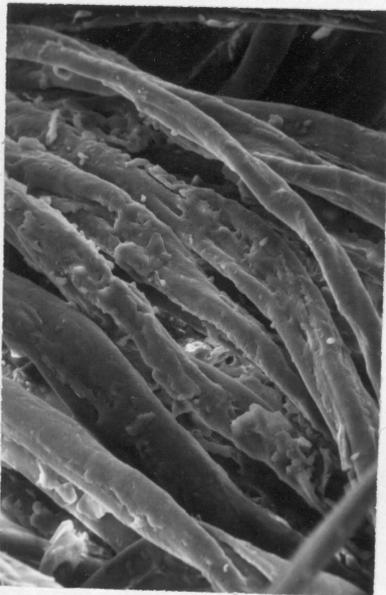


Fig. 7.--Bath II sample  
laundered 5 times in  
hard water with soap  
(375X)

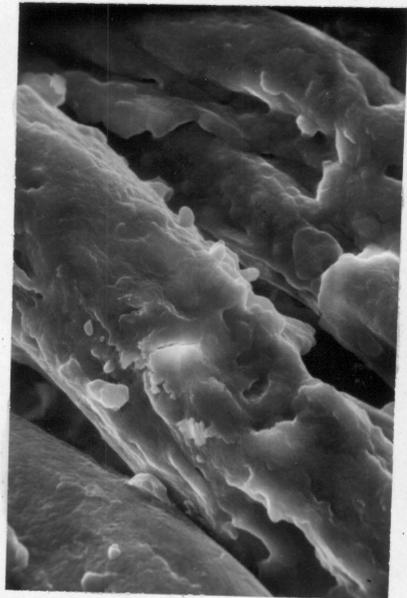


Fig. 8.--Bath II sample  
laundered 5 times in  
hard water with soap  
(1500X)

completely removed and a build up similar to that observed on the Bath I samples after five launderings was shown (Figures 9 and 10).

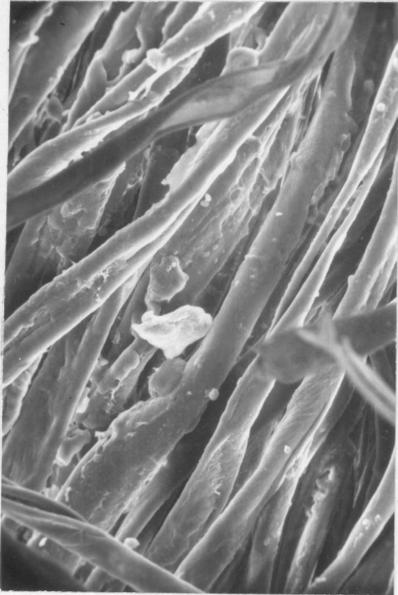


Fig. 9.--Bath II sample  
laundered 10 times in  
hard water with soap  
(375X)

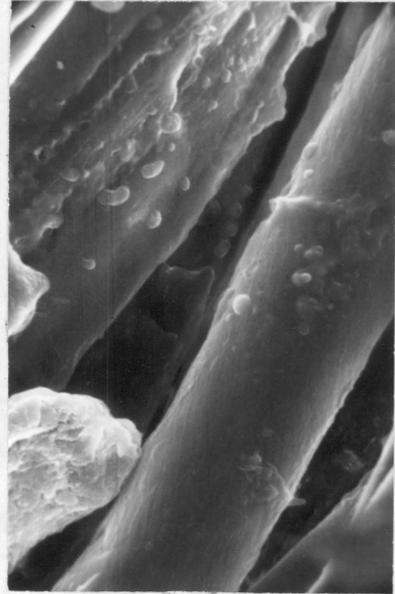


Fig. 10.--Bath II sample  
laundered 10 times in  
hard water with soap  
(1500X)

## CHAPTER V

### CONCLUSIONS

The objectives of this research were to determine the ability to include a chelating agent in a flame retardant finish without altering the nonflammable properties and to determine the effect of the chelating agent on flame retardant fabrics washed in hard water with soap.

Data was obtained by using the test standard for children's sleepwear as stated in DOC FF 3-71 with the following exceptions: all samples were burned in groups of three instead of five and in the warp direction only instead of warp and filling directions; the samples referred to on page 19 were conditioned to  $65 \pm 2\%$  relative humidity prior to burning. Photomicrograph analysis was obtained from pictures taken on a scanning electron microscope.

The data indicated the following major findings:

1. CMC has an effect on the flammability of flame retardant fabrics after being laundered five times with soap. The CMC improved the flame retarding qualities.
2. After ten launderings, the CMC treated samples began to burn the entire test strip length. The

photomicrographs appear to show that the CMC had been removed and the fibers were beginning to accumulate insoluble salt deposits.

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THE INCORPORATION OF A CHELATING AGENT INTO A FLAME  
RETARDANT FINISH ON A COTTON FLANNELETTE AND THE  
EVALUATION OF SELECTED FABRIC PROPERTIES

by

Carolyn Diane Poli

(ABSTRACT)

A 100% cotton flannelette fabric was used to evaluate the following objectives: 1) to determine the ability to include a chelating agent in a flame retardant finish without altering the non-flammable properties; and 2) to determine the effect of the chelating agent on flame retardant fabrics washed in hard water with soap.

Major findings of the research include the following: 1) carboxymethyl cellulose improved the non-flammable qualities; and 2) the carboxymethyl cellulose seemed to wear off after 10 launderings at which time calcium and magnesium deposits were believed to begin accumulation.