TEXTILE FLAME RETARDANTS FROM HOME PRODUCTS

by

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INTRODUCTION

The early history of textile flame retardants involves largely nondurable, water-soluble retardants. One of the first references of a textile fire retardant in the scientific literature is mentioned by Lyons (4). The recipe, formulated in 1735, involves borax, alum, vitriol (a metallic sulfate), or copperas (iron sulfate). Phosphate also was used in early flame retardants for cotton and linens in the work of Gay Lussac in the 1820's (4). The Scientific American of July 27, 1861, noting the death of the wife of Henry Wadsworth Longfellow from a clothing fire recommended the use of "tungstate of soda" or the "phosphate of ammonia" for the treatment of cotton textile items in the home (5).

In the study of water soluble, nondurable flame retardants, borax is the compound that has received the most recent attention. In 1930, the work of Ramsbottom and Snoad demonstrated that a mixture of borax and boric acid provided an excellent flame retardant when applied to cotton textiles. More recently, a 9.5 percent borax and boric acid solution was shown to have a limiting oxygen index (LOI) superior to a commercial flame retardant, tetrakis hydroxymethyl phosphonium chloride (THPC) (3). Phosphate compounds, usually in the form of ammonium phosphate are currently recommended by the United States Department of Agriculture (USDA) for application to cellulosic textiles in the home (6). Phosphate-derived compounds such as THPC are currently used to meet the Department of Commerce (DOC) Standard DOC FF 3-71 for children's sleepwear and are required to meet certain laundry standards for fastness, a condition that water-soluble borax and phosphate compounds cannot meet.

This study was planned to examine the effectiveness of a commercial grade borax and a commercial phosphate (Calgon) in producing flame retardancy in all-cotton flannelette when those chemicals are combined with common household chemicals. The household chemicals, alum (aluminum ammonium sulfate), tartar (potassium tartrate), sal soda (sodium carbonate) and boric acid were chosen for the formulations to be studied because preliminary investigation demonstrated that these chemicals had retardancy action, and all of them, with the exception of tartar, had a past history of use as a flame retardant when combined with other compounds or when used alone. Solutions of borax and each of the following were made: tartar, alum and sal soda, whereas Calgon was mixed with boric acid and tartar. The solutions were applied to a dry all-cotton flannelette by two methods; dipping the fabric into the solution, and by sprinkling the fabric with the solution.

MATERIALS AND METHODS

Fabric

A roller-print flannelette (Valtex Fabrics, New York) was obtained commercially. The physical parameters were as follows:

1. Thread count: Warp, 45 yarns per inch and Fill, 43 yarns per inch
2. Thickness: 0.015 inches at 1 lb. per sq. in. pressure
3. Weight: 3.72 oz. per sq. yd.

A 36 x 44-inch sample of the fabric was used for each exposure of the chemicals to the fabric

Chemical Treatments

For this study, the following concentrations were applied to the dry test fabric using the code at the left margin:

Borax (sodium tetraborate) and Tartar (potassium tartrate)

1a: 10 percent solution, 5 parts borax and 5 parts tartar
1b: 8 percent solution, 3 parts borax and 5 parts tartar
1c: 8 percent solution, 5 parts borax and 3 parts tartar

Borax (sodium tetraborate) and Soda (sodium carbonate)

2a: 10 percent solution, 5 parts borax and 5 parts soda
2b: 8 percent solution, 3 parts borax and 5 parts soda
2c: 8 percent solution, 5 parts borax and 3 parts soda

Borax (sodium tetraborate) and Alum (aluminum ammonium sulfate)

3a: 10 percent solution, 5 parts borax and 5 parts alum
3b: 8 percent solution, 3 parts borax and 5 parts alum
3c: 8 percent solution, 5 parts borax and 3 parts alum

Phosphate (sodium hexametaphosphate) and Boric Acid

4a: 10 percent solution, 5 parts phosphate and 5 parts boric acid
4b: 8 percent solution, 3 parts phosphate and 5 parts boric acid
4c: 8 percent solution, 5 parts phosphate and 3 parts boric acid

Phosphate (sodium hexametaphosphate) and Tartar (potassium tartrate)

5a: 10 percent solution, 5 parts phosphate and 5 parts tartar
5b: 8 percent solution, 3 parts phosphate and 5 parts tartar
5c: 8 percent solution, 5 parts phosphate and 3 parts tartar

Each of the above chemical formulations were dissolved in moderately hard water (approximately 100 ppm total hardness) obtained directly from the tap source at approximately 70°F. . A 36 x 44-inch sample of the test fabric was immersed in each solution for three minutes. Following immersion, the fabric was run through a hand wringer to remove excess moisture and allowed to dry at room temperature. Fabric samples of the identical dimensions were placed on a nonporous surface and sprinkled with 250 milliliters (ml) of the test chemicals using a plastic 500 ml bottle and a sprinkling head commonly used for sprinkling clothing for ironing. Following sprinkling, the fabric samples were folded into a rectangle roughly four inches square and the solution was allowed to wick throughout the fabric for one hour. Then, the fabric was unfolded and allowed to dry at room temperature.

Flame Resistance

Following drying, the treated fabrics were exposed to the American Association of Textile Chemists and Colorists (AATCC) test method 34-1969 for determining the residual flame time, afterglow and char length of the fabric sample (1).

Physical Testing

Before and after application of the finishes, the test fabric underwent physical analysis using methods specified by the American Society for Testing and Materials (ASTM) (2) for:

1. Breaking Strength: ASTM Method D- 1682 64 (1-inch ravelled strip) for determining the pounds of breaking strength
2. Weight: ASTM Method D-1910 64 for determining the ounces per square yard
3. Yarn Count: ASTM Method D-1910 64 for determining the threads per inch, warp and fill
4. Thickness: ASTM Method D-1777 64 for determining the thickness in thousandth of an inch.

Subjective Analysis: Hand

The hand of 36 x 12-inch samples of the fabric, before and after application of the finishes was rated by a five member panel utilizing a linear rating scale from zero to four with zero (0) representing soft and four (4) representing harsh. The scale was printed on 3 x 5-inch cards and each panel member had a separate card for each sample fabric. Before the panel examination the following adjectives were ascribed to the endpoints: SOFT; flexible, downy, smooth, velvety, refined.

HARSH; rigid, stiff, rough, gritty, unrefined. The panel members were asked to make a mark on the linear scale near where they would rate the fabric. The scale was four inches long and a rating near zero corresponded to a soft hand whereas a rating near four indicated that the panel member felt the fabric harsh. The ratings were taken by measuring the distance from zero and recording the ratings to the nearest tenth of an inch.

Statistical Analysis

Data for three second ignition; residual flame times, breaking strength and hand were subjected to 4-way and 3-way analysis of variance, respectively. The flammability results of the afterglow and char length were not analysed statistically as a result of the uniformity of results for both three and twelve second ignition times.

RESULTS

Borax and Tartar

The borax/tartar treatments were effective in preventing residual flame after removal of the flame source. At the 5:5 ratio, the sample extinguished at both 3 and 12 second ignition. The 3:5 and 5:3 ratios proved effective in all laboratory analysis at 12 second ignition, but showed some residual flame time at the 3 second ignition (Table 1). The treated specimens exposed to the 3 second ignition had an overall probability level of 99 percent for all ratios and treatments (by AOV, F test). The data accumulated with the 12 second ignition did not lend itself to statistical analysis. Afterglow on each sample continued after the residual flame was extinguished and the afterglow consumed the entire length of the 10-inch sample. Therefore, for all ratios, char is the full

Table 1. Average Residual Flame Times (in seconds) for Flannelette with Flame Retardant Chemicals Applied by the Dip and Sprinkle Methods for the Three and Twelve Second Ignition Times

Treatments and Ratios	Three Second				Twelve Second			
	Dip		Sprinkle		Dip		Sprinkle	
	Warp	Fill	Warp	Fill	Warp	Fill	Warp	Fill
1a	0	0	0	0	0	0	0	0
1b	0.9	3.2	0.3	1.6	0	0	0	0
1c	0	0.5	3.5	9.2	0	0	0	0
2a	4.1	2.4	0.3	5.1	0	0	0	0
2b	3.5	3.4	7.0	5.2	0	0	0	0
2c	5.9	7.0	5.8	10.6	0	0	0	0
3a	22.5	23.8	20.8	22.3	17.7	13.9	11.2	11.5
3b	19.1	22.1	19.0	21.3	8.2	10.9	7.5	10.1
3c	20.1	17.1	18.8	19.3	9.8	10.4	10.5	10.2
4a	16.1	11.8	17.5	18.1	0	0	0	0
4b	20.8	22.9	29.2	35.7	19.0	0	8.6	6.2
4c	21.3	19.9	20.9	25.1	17.9	0	10.5	0
5a	6.0	5.7	17.1	15.4	0.6	0	2.7	7.9
5b	5.9	3.9	19.2	17.3	0	0	5.0	7.4
5c	14.6	19.8	15.7	15.1	9.2	9.0	3.3	6.2
Control	16.7	16.0			4.9	12.1		

Treatments:

1. Borax/tartar
2 Borax/soda
3. Borax/alum
4. Phosphate/boric acid
5. Phosphate/tartar

Ratios:

a. 10%, 5:5
b. 8%, 3:5
c. 8%, 5:3

Table 2. Average Afterglow (in seconds) for Flannelette with Flame Retardant Chemicals Applied by the Dip and Sprinkle Methods

Treatments and Ratios	Three Second Ignition				Twelve Second Ignition			
	Dip		Sprinkle		Dip		Sprinkle	
	Warp	Fill	Warp	Fill	Warp	Fill	Warp	Fill
1a	>180	>180	>180	>180	>180	>180	>180	>180
1b	>180	>180	>180	>180	>180	>180	>180	>180
1c	>180	>180	>180	>180	>180	>180	>180	>180
2a	>180	>180	>180	>180	>180	>180	>180	>180
2b	>180	>180	>180	>180	>180	>180	>180	>180
2c	>180	>180	>180	>180	>180	>180	>180	>180
3a	BEL[1]	BEL	BEL	BEL	BEL	BEL	BEL	BEL
3b	BEL	BEL	BEL	BEL	BEL	BEL	BEL	BEL
3c	BEL	BEL	BEL	BEL	BEL	BEL	BEL	BEL
4a	BEL	BEL	BEL	BEL	0.5	3.2	0	1.7
4b	BEL	BEL	BEL	BEL	BEL	2.7	BEL	1.0
4c	BEL	BEL	BEL	BEL	BEL	3.3	BEL	2.8
5a	>180	>180	>180	>180	>180	>180	>180	>180
5b	>180	>180	>180	>180	>180	>180	>180	>180
5c	BEL	BEL	BEL	BEL	BEL	BEL	BEL	BEL
Control	BEL	BEL			BEL	BEL		

Treatments:

1. Borax/tartar
2. Borax/soda
3. Borax/alum
4. Phosphate/boric acid
5. Phosphate/tartar

Ratios:

a. 10%, 5:5
b. 8%, 3:5
c. 8%, 5:3

[1] Burned Entire Length

Table 3. Average Breaking Strength (in pounds) of Flannelette Treated With Flame Retardant Chemicals by both the Dip and Sprinkle Methods When Analysed on the Constant-Rate-of-Extension Tester

Treatments and Ratios	Dip		Sprinkle	
	Warp	Fill	Warp	Fill
1a	18.3	22.1	18.3	24.3
1b	20.4	18.3	21.3	17.9
1c	18.4	19.4	19.1	20.8
2a	19.4	22.6	18.7	22.4
2b	22.2	18.4	17.8	19.5
2c	18.5	19.8	19.0	23.7
3a	21.8	26.7	19.0	27.5
3b	23.3	20.4	22.9	20.9
3c	20.4	24.5	20.0	24.2
4a	17.0	19.6	18.4	22.9
4b	19.8	18.1	19.6	18.4
4c	17.7	21.1	18.6	20.4
5a	19.5	26.2	19.3	25.5
5b	24.0	19.6	20.2	17.6
5c	19.6	27.6	19.5	25.4
Control	22.8	19.0		

Treatments:

1. Borax/tartar
2. Borax/soda
3. Borax/alum
4. Phosphate/boric acid
5. Phosphate/tartar

Ratios:

a. 10%, 5:5
b. 8%, 3:5
c. 8%, 5:3

Table 4. Average Ratings of the Hand of Flannelette Treated with the Flame Retardant Chemicals by both the Dip and Sprinkle Methods

Treatments and Ratios	Ratings[1] Dip	Sprinkle
1a	1.8	0.8
1b	0.9	1.6
1c	1.9	1.4
2a	2.3	2.6
2b	2.5	2.6
2c	1.9	1.7
3a	2.2	1.8
3b	2.6	1.1
3c	1.9	1.6
4a	3.0	2.7
4b	2.9	1.9
4c	1.4	2.9
5a	1.8	1.3
5b	1.0	1.2
5c	2.1	1.1
Control	0.5	

Treatments:

1. Borax/tartar
2. Borax/soda
3. Borax/alum
4. Phosphate/boric acid
5. Phosphate/tartar

Ratios:

a. 10%, 5:5
b. 8%, 3:5
c. 8%, 5:3

[1] Zero (0) = soft, Four (4) = harsh

length of the sample when the afterglow exceeds 180 seconds indicating in all cases, the afterglow was non-extinguishing (Table 2).

Breaking strengths of the fabrics treated with borax/tartar, 5:5 and 3:5 ratios, were approximately 4 pounds lower than the fabric as-purchased which had a breaking strength of 22.8 pounds in the warp, whereas the fill was unaffected. The probability level for all the treatments and ratios of the treated specimens was 99 percent.

The hand of the fabrics, borax/tartar treated, was rated by the panel at 1.8 for the 5:5 ratio; the dipped samples, and 0.8 for the sprinkled application. The 3:5 ratio was rated at 0.9 for dipped and 1.6 for sprinkled. The 5:3 ratio gave the highest ratings with scores of 1.9 for dipped and 1.4 for sprinkled. The as-purchased fabric rated by the panelists gave a score of 0.5, a zero being the lowest possible rating. Hand ratings gave no probability level greater than 95 percent although the untreated fabrics were rated softer than the treated samples in all cases.

Borax and Soda

The borax/soda treatments were somewhat effective in restricting residual flame time (Table 1). The samples would ignite but the residual flame would extinguish rapidly following removal of the flame source. The 5:5 ratio of the treatment was the most effective in terms of seconds of flaming for both dipped and sprinkled fabrics. With the 3 second ignition, the 3:5 ratio gave 3.5 and 3.4 second residual flame times in warp and fill, respectively; for the dipped method, and 7.0 and 5.2 seconds for warp and fill, respectively; for the sprinkled samples. The 5:3 ratio, dipped, at 3 seconds ignition gave 5.9, warp and 7.0, fill; and warp, 5.8 and the fill, 10.6 for the sprinkled samples. The overall probability level for all treatments and ratios combined was 99 percent. The 12 second ignition time exhibited no residual flame time in all ratios, warp and fill. Afterglow continued after the flame was extinguished and consumed the entire sample length. All times for afterglow were greater than 180 seconds (Table 2).

The breaking strength ratings of the borax/soda treated samples followed the examples of the borax/tartar treatments and was lower in the warp direction and greater in the fill when compared to the as-purchased (control) samples (Table 3). The dip or sprinkle methods had little effect on the breaking strength of the samples. The 5:5 and 5:3 ratios seemed to have the greatest effect on breaking strength, whereas the 3:5 ratio had little effect. The 5:5 ratio gave a breaking strength of 19.4 pounds, warp; and the 5:3 ratio gave 18.5 pounds, warp, compared to the as-purchased at 22.8 pounds in the warp direction. Similar to the borax/tartar treatments, the fill seemed strengthened rather than weakened by the borax/soda treatments. The method of application had no effect on breaking strength.

The samples treated with borax/soda had a 2.3 hand rating for the 5:5 ratio, a 2.5 rating for the 3:5 and 1.9 for the 5:3 ratio with the dip method. For the sprinkled, the 5:5 had a 2.6, the 3:5, a 2.6 and for the 5:3, a 1.7 for that ratio. There was no significant probability level attached to the method of application in affecting the hand of the samples.

Borax and Alum

The fabric samples treated with the borax/alum finish ignited and burned their entire length (BEL) in every case, regardless of the ratio and method of application. The treatments did not show any indication of flame retardancy. All ratios of borax/alum had no significant effect on fabric breaking strength in the warp and fill directions. The panelists assigned hand scores to the borax/alum specimens as follows: 2.2 for the 5:5 ratio, 2.6 for the 3:5 and 1.9 for the 5:3 ratio for the dipped samples. For the sprinkled samples, the 5:5 ratio was 1.8, the 3:5, 1.1 and the 5:3, 1.6. The control fabric was rated 0.5 for hand.

Phosphate and Boric Acid

All samples given phosphate/boric acid treatments ignited under 3 second ignition regardless of method of application and burned the entire length of the sample. Twelve second ignition times, however, produced small residual flame times, and in the case of 12 second ignition, fill samples showed that the treatments were self-extinguished (Table 1). The treatment ratio 5:5 produced fabric that was flame retardant in both warp and fill directions. The dipped samples had the lowest residual flame ratings in the 3 second ignition times. The difference in the methods of application was significant at the 99 percent level. The 3:5 ratio burned the entire length of the sample with the exception of the fill sample under 12 second ignition. The 5:3 ratio gave similar results with the 12 second ignition fill sample giving a residual flame time of zero for both dip and sprinkle methods of application.

The phosphate/boric acid samples were the only samples tested that did not allow an afterglow to consume the entire sample length. For the 12 second ignition time samples that did not ignite, the afterglow was less than 3.5 seconds (Table 2). For those fill samples that did not ignite, the samples treated with the dip method had a char length no greater than 3.8 inches, whereas the sprinkled samples at a 5:5 ratio had a 3.3 inch char, the 3:5 ratio, a 7.1 inch char and the 5:3, a 4.1 inch char length.

The phosphate/boric acid treatment appeared to have the most deleterious effect on warp fabric strength. The 5:5 ratio lowered the warp breaking strength to 17.0 pounds, the 3:5 to 19.8 and the 5:3 ratio to 17.7 pounds. The as-purchased samples had a breaking strength of 22.8 pounds in the warp direction. The fill samples in each treatment ratio were unaffected and often strengthened when compared with the control (Table 3). The probability level for the ratios' effect on the breaking strength of the samples was 99 percent. There was no significant difference in the breaking strengths between the methods of application.

The 5:5 ratio, dipped, gave the harshest hand, a 3.0, the 3:5, a 2.9 and the 5:3, a 1.4. For the sprinkled ratios, the 5:5 ratio gave a 2.7, the 3:5, a 1.9 and 2.9 for the 5:3 ratio compared to the control at 0.5.

Phosphate and Tartar

The ratios of 5:5 and 3:5 phosphate/tartar were flame retardant to a degree, although afterglow consumed the remaining samples after the residual flame had extinguished. The 3 second ignition time produced residual flame times of 6.0 seconds for the warp samples and 5.7 seconds for the fill with the 5:5 ratio, dip method. The 3:5 ratio gave 5.9 seconds for warp and 3.9 for fill, dip method (Table 1). The sprinkled samples gave higher residual flame times than previous samples indicating a higher degree of retardancy. The 5:3 ratio applied by both methods burned the entire length at both 3 and 12 second ignition times. There was no ignition for the 5:5 and 3:5 ratios at the 12 second ignition with the chemicals applied by the dip method, whereas all the sprinkled samples ignited, although the flame extinguished within 7.9 seconds.

The breaking strength of the fabrics treated with the 5:5 and 5:3 ratios was lessened in the warp direction, and generally somewhat increased in the fill regardless of the method of application. Probability levels of the effect of the finishes on breaking strength was at the 99 percent level. The 3:5 ratio had the lesser effect on breaking strength giving a breaking strength of 24.0 pounds for warp and 19.6 pounds for fill. This is compared to the as-purchased samples giving a 22.8 pound breaking strength for warp and 19.0 for fill. There was no significant difference between method of application and their effect on breaking strength.

The hand ratings for this group of samples was lower than the average ratings for the previous samples although there was no statistically significant probability level. The 5:5 ratio was rated by the panelists at 1.8, the 3:5 at 1.0 and the 5:3 ratio at 2.1 for dipped samples. Although samples sprinkled with the treatments were rated somewhat lower than dipped specimens, there was no significant difference between methods of application.

DISCUSSION

Flammability

Of all the treatments examined, only the 5:5 ratio of borax/tartar gave a consistant zero residual flame time. Other treatments such as all ratios of borax/tartar, borax/soda and the 5:5 and 3:5 ratios of phosphate/tartar had small residual flame times, but were subject to excessive afterglow that consumed the entire sample. With borax/tartar, borax/soda, phosphate/boric acid and the 5:5 and 3:5 ratios of phosphate/

tartar, there were samples that gave zero residual flame times but only for the fill samples exposed to a 12 second ignition. The warp direction often ignited, whereas the fill usually would not support a flame. This might be attributed to the more bulky yarns found in the fill or the greater density of the warp yarn lattice the flame has to consume. The differences between residual flame times for the 3 and 12 second ignitions was found by Weaver (8) to be related to add-on concentrations. Weaver found that with some ammonium and phosphate flame retardants, there is a seemingly "critical" area where a small increase in the chemical add-on can enable a cotton twill fabric to pass a formerly failed 3 second ignition test. This phenomena may be involved in the case of phosphate/boric acid and phosphate/tartar specifically (Tables 1 and 2). The critical factors of the add-on concentrations may influence a sample to ignite in the warp but not in the fill, or with a 3 second ignition, but resist flame with the 12 second ignition time.

The borax/alum and the 5:3 ratio of the phosphate/tartar treatment showed no signs of retardancy. The afterglow of every sample, except the three ratios of phosphate/boric acid, was excessive, and if the fabric was not consumed by flame, afterglow proceeded after the flame extinguished to consume the entire sample and give recorded char lengths of 10 inches. The phsophate/boric acid ratios that controlled afterglow likely could be attributed to the presence of boric acid because phosphate also was present in the phosphate/tartar, but afterglow was as damaging as in the borax-based finishes.

The borax/tartar, 5:5 ratio, had the greatest effectiveness in preventing residual flame time, but allowed excessive afterglow. The most promising finishes, the phosphate/boric acid concentrations, were of the greatest value in preventing afterglow and in the case of 12 second ignition samples from the fill direction, prevented residual flame. Perhaps an increase in concentration of the phosphate/boric acid solutions at the 5:5 ratio would allow the flannelette to pass both 3 and 12 second ignition times. Borax/tartar meets this requirement, but afterglow is not controlled.

The dip method of application provided the greatest amount of retardancy in terms of residual flame time (Table 1). Add-ons are more easily controlled by dipping and a greater evenness and/or spread of the retardancy chemicals may be expected than by sprinkling the chemical over the fabric with a bottle and sprinkling head.

The Department of Commerce standards for children's sleepwear (DOC FF 3-71) (7) provides a textile flammability "acceptance criterion" as follows:

1. The average char length of five specimens cannot exceed 7.0 inches.
2. No individual specimen can have a char length of 10.0 inches.
3. No individual specimen can have a residual flame time greater than 10.0 seconds.

Although DOC FF 3-71 requires specimens to be tested in a state of dryness, while the AATCC test method specifies conditioned specimens at 70° F. and 65° relative humidity. It may be seen from data in tables 1 and 2, that no single ratio of any of the treatments met this criterion. The 5:5 ratio of borax/tartar met the residual flame requirements, but fails the char length specifications because the uncontrolled afterglow resulted in a 10.0 inch char length in all samples. The treatment that most closely approximated the overall criteria of DOC FF 3-71; the 5:5 ratio of phosphate/boric acid, met the residual flame and char length requirements with an average char length of 3.8 inches for both dipped and sprinkled samples. This, unfortunately, is only with the exposure of the flannelette sample to the 12 second ignition time, whereas the sample exposed to the 3 second ignition burns the entire length, thus failing the acceptance criterion number three as listed above. Again, if the critical add-on concentration is applicable with phosphate/boric acid, this finish, although water-soluble, and not laundry fast, may find a domestic use.

Breaking Strength

The treated samples, borax/alum had the least effect on breaking strength, but had the poorest flammability rating of the treatments examined. The most promising finish that combined a degree of flame retardancy and controlled afterglow had the greatest effect on lowering breaking strength in the warp direction. The 5:5 ratio of phosphate/boric acid lowered the warp breaking strength a full 5 pounds, the fill direction remaining unaffected. The significant differences in treatment and ratio interactions were at the 99 percent level. The method of application had no significant effect on breaking strength. The 5:5 and 5:3 ratios in all the treatments consistantly appeared to have the greatest effect on altering breaking strengths (Table 3). One might expect the 10 percent concentrations, 5:5 ratios, to have the greatest effect on breaking strengths because of the greater concentrations, but this was not the case. The borax/tartar and borax/soda ratios that gave the lesser residual flame times had a smaller effect on breaking strengths than did the samples treated with the phosphate/boric acid, which cannot be attributed to the low pH of the boric acid (5.3) as the tartar gave a pH of 4.7 and the alum, a pH of 4.5, both lower than the pH of boric acid.

The increase in the fill-direction breaking strength in virtually every case of ratio and treatment cannot be related to fabric shrinkage alone as thread counts performed on each treated sample revealed a consistant 44 yarns per inch (ypi) in the fill direction as compared to the as-purchased ypi of 43. There could have been a constricting or drawing up of fill yarns that resulted in the 47-48 ypi in the treated fabrics as compared with the control ypi of 45.

Hand

The finish with the greatest promise proved to be the most severe in altering hand ratings (Table 4). The 5:5 ratio of phosphate/boric acid gave a hand rating of 3.0 as compared to the control fabric at 0.5. The borax/tartar treatments seemed to have the lesser effect on hand although no significant difference was found between treatments or ratios. There was also no significant probability level between methods of application.

SUMMARY

Household chemicals: borax/tartar, borax/soda, borax/alum, phosphate/boric acid and phosphate/tartar were dissolved in water and applied to all-cotton flannelette in three ratios; 5:5 (10%), 3:5 (8%) and 5:3 (8%), the ratios following the listing of the chemicals. Each of the fifteen solutions were applied to the flannelette by two methods; dipping and sprinkling. The treated fabrics were tested for flammability and breaking strength to determine any deleterious effects of the treatments and other selected physical parameters including hand. Of the chemicals analysed, borax/tartar at a 5:5 ratio provided the greatest degree of flame retardancy and would not ignite under 3 or 12 second ignition times.

With this treatment, however, excessive afterglow was noted that eventually consumed the 10-inch test sample. Other formulations that gave a degree of flame retardancy were borax/soda, which would not allow flaming beyond 10.6 seconds in all ratios but had excessive afterglow, phosphate/tartar, which would not ignite and sustain a flame with the 5:5 and 3:5 ratios but burned completely at the 5:3 ratio and phosphate/boric acid which consistantly ignited at 3 seconds but showed little residual flame at a 12 second ignition time and suppressed afterglow in less than 3.3 seconds. It is thought that a greater concentration of phosphate/boric acid may produce a flame retardant that passes both 3 and 12 second ignition and controls afterglow. The formulations of borax/alum burned completely at every ratio and gave no indication of retardancy.

Breaking strengths were affected largely in the warp direction with the exception of the ratios of borax/alum. The formulation of phosphate/boric acid had the greatest weakening effect. The borax/tartar, borax/soda and phosphate/tartar ratios had a relatively moderate effect on warp breaking strength lowering the warp breaking strength approximately 4 pounds. The fill direction samples were largely unaffected and in some cases, strengthened.

The hand of the treated samples was rated harshest when treated with phosphate/boric acid and lowest when treated with phosphate/tartar. The borax-based formulae had a moderate effect on panel ratings, which were designed to judge hand on a scale ranging from soft to harsh.

Further work needs to be performed especially with the more promising phosphate/boric acid treatment to determine the effect of higher add-ons on residual flame times. The flame resistant but non-glow resistant borax/tartar also holds some promise provided that additives that limit afterglow, the weakness of this treatment, can be discovered. The other chemical combinations investigated such as borax/alum, borax/soda and phosphate/tartar are of marginal value and hold little promise.

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APPENDIX

Table 5. Analysis of Variance for Residual Flame Time of Flannelette Treated with Flame Retardant Chemicals for Three Second Ignition Time

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F-Value
Method of Application	1	556.320	556.320	59.605**
Treatment	4	15463.429	3865.857	414.194**
Warp & Fill	1	54.731	54.721	5.862*
Ratio of Chemicals	2	425.761	212.880	22.808**
Treatment x Method of Application	4	504.162	126.040	13.504**
Warp & Fill x Method of Application	1	39.690	39.690	4.252
Method of Application x Ratio of Treatments	2	118.539	59.269	6.350*
Treatments x Warp & Fill	4	29.924	7.231	0.774
Treatments x Ratio	8	1188.350	148.543	15.915**
Ratio x Warp & Fill	2	32.375	16.187	1.734
Method of Application x Warp & Fill x Treatment	4	79.956	19.988	2.141
Method of Application x Treatment x Ratio	8	697.505	87.188	9.341**
Method of Application x Warp & Fill x Ratio	2	14.585	7.292	0.781
Treatment x Warp & Fill x Ratio	8	166.456	20.807	2.229
Treatment x Warp & Fill x Ratio x Method of Application	8	74.667	9.333	0.425
Error	180	3946.720	21.926	
Total	239	23382.187		

* Significant at 95% Level

** Significant at 99% Level

Table 6. Average Char Length (in inches) of Ten-inch Flannelette Samples with Flame Retardant Chemicals Applied by the Dip Method

Treatments and Ratios	Three Second Ignition		Twelve Second Ignition	
	Warp	Fill	Warp	Fill
1a	10.0	10.0	10.0	10.0
1b	10.0	10.0	10.0	10.0
1c	10.0	10.0	10.0	10.0
2a	10.0	10.0	10.0	10.0
2b	10.0	10.0	10.0	10.0
2c	10.0	10.0	10.0	10.0
3a	10.0	10.0	10.0	10.0
3b	10.0	10.0	10.0	10.0
3c	10.0	10.0	10.0	10.0
4a	10.0	10.0	3.9	3.4
4b	10.0	10.0	10.0	3.8
4c	8.3	10.0	10.0	3.8
5a	10.0	10.0	10.0	10.0
5b	10.0	10.0	10.0	10.0
5c	9.3	10.0	10.0	10.0
Control	10.0	10.0	10.0	10.0

Treatments:

1. Borax/tartar
2. Borax/soda
3. Borax/alum
4. Phosphate/boric acid
5. Phosphate/tartar

Ratios:

a. 10%, 5:5
b. 8%, 3:5
c. 8%, 5:3

Table 7. Average Char Length (in inches) of Ten-inch Flannelette Samples with Flame Retardant Chemicals Applied by the Sprinkle Method

Treatment and Ratios	Three Second Ignition		Twelve Second Ignition	
	Warp	Fill	Warp	Fill
1a	10.0	10.0	10.0	10.0
1b	10.0	10.0	10.0	10.0
1c	10.0	10.0	10.0	10.0
2a	10.0	10.0	10.0	10.0
2b	10.0	10.0	10.0	10.0
2c	10.0	10.0	10.0	10.0
3a	10.0	10.0	10.0	10.0
3b	10.0	10.0	10.0	10.0
3c	10.0	10.0	10.0	10.0
4a	10.0	10.0	3.8	3.3
4b	10.0	10.0	10.0	7.1
4c	10.0	10.0	8.7	4.1
5a	10.0	9.0	10.0	10.0
5b	10.0	10.0	10.0	10.0
5c	10.0	10.0	10.0	10.0
Control	10.0	10.0	10.0	10.0

Treatments:

1. Borax/tartar
2. Borax/soda
3. Borax/alum
4. Phosphate/boric acid
5. Phosphate/tartar

Ratios:

a. 10%, 5:5
b. 8%, 3:5
c. 8%, 5:3

Table 8. Analysis of Variance of Breaking Strength of Flannelette Treated with Flame Retardant Chemicals by both the Dip and Sprinkle Method when Analysed of the Constant-Rate-of-Extension Tester

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F-Value
Method of Application	1	0.196	0.196	0.031
Treatment	4	599.655	149.913	24.279**
Warp & Fill	1	403.649	403.649	65.373**
Ratio of Treatment	2	127.811	63.905	10.349**
Method of Application x Treatment	4	68.932	17.233	2.790
Method of Application x Warp & Fill	1	24.025	24.025	3.891
Method of Application x Ratio	2	28.832	14.416	2.334
Treatment x Warp & Fill	4	58.029	14.507	2.349
Treatment x Ratio	8	69.425	8.678	1.405
Warp & Fill x Ratio	2	895.345	447.672	72.502
Method of Application x Treatment x Warp & Fill	4	31.082	7.770	1.157
Method of Application x Treatment x Ratio	8	62.869	7.858	1.170
Method of Application x Warp & Fill x Ratio	2	10.805	5.402	0.875
Treatment x Ratio x Warp & Fill	8	128.245	16.030	2.596
Method of Application x Ratio x Warp & Fill x Treatment	8	49.396	6.174	2.258
Error	300	820.230	2.734	
Total	359	3378.533		

* Significant at 95% Level

** Significant at 99% Level

Table 9. Analysis of Variance of the Average Hand Ratings of Flannelette Treated with Flame Retardant Chemicals by both the Dip and Sprinkle Methods

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F-Value
Method of Application	1	2.457	2.457	1.222
Treatment	4	28.574	7.143	3.554
Ratio of Treatment	2	1.512	0.756	0.376
Method of Application x Treatment	4	3.244	0.811	0.403
Method of Application x Ratio	2	0.758	0.379	0.188
Treatment x Ratio	8	6.520	0.815	0.405
Method of Application x Treatment x Ratio	8	16.077	2.009	3.024
Error	120	79.735	0.664	
Total	149	138.880		

* Significant at 95% Level

** Significant at 99% Level

Table 10. Average Physical Analysis Results of Flannelette Following Flame Retardant Finish Application by the Dip Method

Treatments and Ratios	Fabric Weight (oz. per sq. yd.)	Fabric Thickness (in inches)	Thread Count (yarns per inch) Warp	Fill
1a	4.22	0.015	48	44
1b	4.08	.015	47	44
1c	4.23	.015	48	44
2a	4.52	.015	47	44
2b	4.18	.016	47	44
2c	4.14	.016	47	44
3a	4.20	.017	48	44
3b	4.20	.015	48	44
3c	4.24	.016	48	44
4a	4.37	.015	48	44
4b	4.00	.015	48	44
4c	4.04	.015	48	44
5a	4.30	.014	48	44
5b	4.24	.015	48	44
5c	4.05	.014	48	44
Control	3.72	0.015	45	43

Treatments:

1. Borax/tartar
2. Borax/soda
3. Borax/alum
4. Phosphate/boric acid
5. Phosphate/tartar

Ratios:

a. 10%, 5:5
b. 8%, 3:5
c. 8%, 5:3

TEXTILE FLAME RETARDANTS FROM HOME PRODUCTS

by

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B.S., California State University, Northridge, 1971

AN ABSTRACT OF A MASTER'S THESIS

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MASTER OF SCIENCE

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Household chemicals; borax/tartar, borax/soda (washing soda), borax/alum, phosphate (Calgon)/boric acid and phsophate/tartar were dissolved in water and applied to dry all-cotton flannelette with the possibility of acting as a flame retardant, in 3 ratios; 5:5 (10%), 3:5 (8%), and 5:3 (8%). The ratios of the chemicals were applied by two methods; dipping and sprinkling. Following treatment, the fabrics were analysed for flammability, breaking strength, hand, and physical parameters to determine any damaging effects of the treatment.

The ratio of 5:5 borax/tartar was the most effective in preventing flame following 3 and 12 second ignition. The 5:5 ratio would not prevent afterglow. The ratios of borax/alum gave no indication of flame retardancy and the ratios of borax/soda held residual flame time to under 10.6 seconds but would not prevent afterglow. The ratios of 5:5 and 3:5 phosphate/tartar succeeded in preventing flaming but also had uncontrollable afterglow. The 5:5 ratio of phosphate/boric acid was the most effective and meets most closely, the requirements for a flame retardant finish by limiting char length to under 7.0 inches and suppressing afterglow and residual flame time. Phosphate/boric acid would not pass the 3 second ignition test, however, possible because of marginal add-on of the finish.

All fabric breaking strengths were affected by the chemical in the warp samples with the exception of borax/alum. Phosphate/boric acid had the greatest weakening effect with borax/tartar, borax/soda and phosphate/tartar having a moderate effect on warp strength. Fill samples remained unaffected and in some cases, strengthened.

Phosphate/boric acid had the harshest effect on hand and phosphate/tartar altered hand the least, other treatments having a medium effect.