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FLAME RESISTANT FINISH FOR TEXTILES

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The invention described herein, if patented, may be manufactured and used by or for the Government for governmental purposes without the payment to us of any royalty thereon.

This invention relates to flame-resistant organic fabrics, which retain their flame resistance after repeated launderings, and to compositions and methods for imparting such flame resistance to fabrics.

It was discovered several years ago that tetrakis hydroxymethyl phosphonium chloride (THPC) has good flame-resistance imparting properties for cotton fabrics and other textiles. It is also known that a combination of THPC and tris-1-aziridinyl phosphine oxide (APO) renders cotton or other fabrics flame resistant.

APO, by itself, when applied to a fabric in an aqueous bath, does not improve the flame resistance of the fabric. We attribute this to the fact that monomeric APO does not sufficiently polymerize. Inasmuch as THPC is a relatively expensive chemical, we have endeavored to develop a flame resistant textile finishing composition which does not contain THPC.

We have now discovered that an aqueous solution of APO when applied to a cotton or other fabric in the presence of diammonium phosphate (DAP) in certain specified proportions, results in a flame resistant fabric which retains its flame resistance even after repeated launderings. Without limiting ourselves to any particular theory underlying this unexpected result, we presently believe that the diammonium phosphate acts as a polymerization catalyst for the APO and enables it to form a polymer in situ on and between the fibers of the fabric, when cured above room temperature.

Inasmuch as diammonium phosphate is a very inexpensive chemical, such a composition is more economical than a composition of THPC and APO as active ingredients.

It is accordingly an object of our invention to provide an economical and efficient flame resistance imparting treatment for fabrics, which can be carried out with conventional textile padding and curing equipment, without noxious fumes and without the need for specially trained personnel.

Another object of our invention is an economical flame resistance imparting textile finish which in its preferred form retains its efficiency after repeated launderings.

Other objects and advantages of our invention will readily occur to the expert from the following description of several preferred methods and compositions for carrying our invention into practice.

Our research has shown that a flame resistant textile finish is provided by an aqueous solution of diammonium phosphate and APO, wherein the APO is present in a proportion of at least 1 to 6 parts by weight per 1 part by weight of diammonium phosphate, the strength of the solution being at least about 5 percent APO. The finish is laundry-fast if the APO concentration is at least about 15 percent. The strength of the solution may be raised above 15 percent APO; however, above 20 percent no appreciable increase in flame resistance and laundry resistance occurs. In a presently preferred embodiment of our invention, the combined aqueous solution contains 5 percent (or more) diammonium phosphate and about

15–20 percent (in a preferred embodiment 18.2 percent) APO.

Padding of the fabric is effected for a substantial wet pick-up, say of the order of 55–75 percent, which after drying and curing corresponds to a dry weight add-on of about 4–15 percent of APO polymer (preferably above 7 percent), depending on the strength of the solution in terms of APO contents.

Where laundry resistance is not of the essence, the APO strength of the bath may be reduced to about 5 percent strength level; however, for laundry-fastness, an APO strength of about 15 percent or more is needed.

It is neither necessary nor desirable that the diammonium phosphate be present in the solution in an amount exceeding that of the APO. To facilitate wet pick-up, a small amount of a commercial wetting agent, as for instance Triton X–100 may be added to the solution; however, this is optional.

After the fabric is removed from the bath, it is dried and cured above room temperature by a one-step or plural-step exposure to an elevated temperature which is non-injurious to the fabric; say drying at about 250° F. and curing of the dried fabric at about 300° F. to produce the APO polymer in situ. Obviously, these two steps can be combined, e.g., by drying and curing step at about 250–300° F.

We now proceed to illustrate the practice of our invention by giving several examples.

EXAMPLE 1

Fifty yards of 8.5 oz. cotton sateen cloth (Military Specification MIL–C–12095) was padded through a bath containing 5% diammonium phosphate and 18.2% APO in the following bath formulation:

	Lbs.
Diammonium phosphate	4.25
Imine IP (73% solids; being a commercial form of APO)	21.25
Triton X–100	0.4
Water	59.1
Total (10 gal.)	85

The wet pick-up was 75%. The fabric was dried at 250° F. for two minutes and cured at 300° F. for five minutes. The weight of the treated fabric, including the resin finish formed in situ thereon was 10% above the weight of the untreated fabric. The treated fabric was subdivided into several identical lengths, some of these identical samples were submitted to laundering at predetermined numbers of cycles. The flame resistance and tear resistance of the treated fabric (unlaundered and laundered) were determined by a standard method, using a vertical Bunsen burner in accordance with Textile Testing Method 5902 (Federal Specification CCC–T–191*b*, Textile Test Methods, dated May 15, 1951). Afterflame and after-glow were measured in seconds, and the char length in inches. The following table summarizes the results obtained:

Table 1

Treated Fabric	After-flame (Seconds)	After-glow (Seconds)	Char Length (Inches)
Initial	0	1.2	3.9
Laundered:			
1 cycle	0	1	4.3
3 cycles	0	1	4.9
6 cycles	0	1	4.6
9 cycles	0	1	4.8
12 cycles	0	1	4.7
15 cycles	0	2	4.9

EXAMPLES 2–6

The following examples illustrate a number of variations of the treatment of Example 1.

Table 2

	Example 2			Example 3			Example 4			Example 5		
	15% APO 2½% DAP			15% APO 5% DAP			15% APO 10% DAP			15% APO 15% DAP		
Wet pick-up percent resin	63.0			63.5			68.3			66.0		
Add-on (dry weight)	7.6			10.5			14.8			13.7		
	AF	AG	CL	AF	AG	CL	AF	AG	CL	AF	AG	CL
Initial	0	1	4.2	0	1	3.8	0	1	3.9	0	1	2.7
Laundered:												
1 cycle	0	2	4.7	0	1	3.9	0	1	4.0	0	1.7	3.1
3 cycles	0	1.3	4.5	0	1	3.8	0	1	4.4	0	1.1	4.0
6 cycles	0	1.2	5.3	0	1	4.5	0	1	4.7	0	1	4.3
9 cycles	0	1.2	5.0	0	1	4.5	0	1	4.7	0	1.4	4.1
12 cycles	0	2.1	6.0	0	1	4.4	0	1	4.4	0	1.0	4.0
15 cycles	0	1.2	4.3	0	1	4.4	0	1.7	4.4	0	1.2	3.7

AF = Afterflame (seconds).
AG = Afterglow (seconds).
CL = Char length (inches).

In a run with a solution of 10% APO and 10% diammonium phosphate for a wet pick-up of 65.5% and a resin add-on (dry weight) of the finished fabric of 10.5%, flame resistance was imparted as follows: after-flame 0 seconds, after-glow 1 second, char length 3.9". This is quiet acceptable. However, after repeated launderings (3 cycles) the flame resistance was lost. A similar result was obtained with a solution containing 10% APO and 5% diammonium phosphate (dry add-on 7%).

In two control experiments with 5% APO, and 5 and 10% diammonium phosphate, respectively (resin add-on dry weight, 4.4 and 6.7%, respectively), initial flame resistance was similar to that imparted by the just-described bath containing 10% APO but the flame resistance did not survive a one-cycle laundering.

While the foregoing examples are in terms of cotton fabrics, the invention can also be practiced with other cellulosic or non-cellulosic fibrous materials such as rayon, wool and the like, to reduce their flammability.

In order to substantiate the synergistic effect of our fabric finishing composition, and to determine whether diammonium phosphate is retained therein before and after laundering, we have applied the same with the use of diammonium phosphate, containing radioactive phosphorus (P^{32}).

An 8.5 oz. cotton sateen fabric was immersed in a bath containing 5% diammonium phosphate only, the phosphorus component of which was radioactive P^{32}. The fabric was squeezed, oven dried at 100° C. (212° F.), and cured for five minutes at 140° C. (284° F). Using a beta-particle counter, it was found that only 6.95% of the radio active diammonium phosphate was retained after four cold water rinses.

The same type of cotton fabric was then immersed in a bath containing 5% radioactive diammonium phosphate and 18% APO. The fabric was oven dried at 100° C. and cured for five minutes at 140° C. After four cold water rinses the fabric was found by the beta-particle counter to contain 43.6% of the original radioactive diammonium phosphate. The fabric was then given four hot water rinses and was found by the same technique to contain 40.5% of the original radioactive diammonium phosphate. After boiling for one-half hour in soap and soda ash (.2% soap and .1% soda ash), the fabric was found by the same technique to have retained 36.6% of the original radioactive diammonium phosphate.

These results indicate that phosphorus from the diammonium phosphate is firmly retained in the APO finish produced in the presence of diammonium phosphate after repeated hot water laundering, while diammonium phosphate, applied by itself, is easily rinsed out by laundering.

Having thus described the principle of our invention, and several methods for carrying the same into practice, we wish it to be understood that specific operating data set forth in such examples are to be taken as illustrative only and not for the purpose of limiting the scope of our invention thereto, inasmuch as a number of variations and modifications will readily ocur to the expert without departing from the spirit of our invention. We thus intend to claim our invention broadly and to that end define the same by the appended claims.

We claim:

1. A flame resistant fibrous organic fabric impregnated with tris-1-aziridinyl phosphine oxide polymerized in situ on said fabric in the presence of diammonium phosphate, said tris-1-aziridinyl phosphine oxide being in a proportion from about 1 to about 6 parts by weight per 1 part by weight of said diammonium phosphate, said tris-1-aziridinyl phosphine oxide and said diammonium phosphate being applied to said fabric from a solution containing at least about 5 percent tris-1-aziridinyl phosphine oxide.

2. A flame resistant fabric according to claim 1, said fabric being impregnated cotton fabric.

3. Flame-resistant fabric according to claim 1, wherein said tris-1-aziridinyl phosphine oxide and said diammonium phosphate are applied to said fabric from a solution containing at least about 15 percent tris-1-aziridinyl phosphine oxide, whereby said fabric retains its flame-resistance after repeated launderings.

4. A laundry-fast frame-resistant fabric according to claim 3, said fabric being impregnated cotton fabric.

5. A flame-resistance-imparting textile finishing composition for fibrous organic fabrics, comprising an aqueous solution of diammonium phosphate and tris-1-aziridinyl phosphine oxide, said tris-1-aziridinyl phosphine oxide being present in at least about 5 percent strength and a proportion of about 1 to 6 parts by weight per 1 part by weight of diammonium phosphate.

6. A laundry-fast composition according to claim 5, wherein the strength of said solution is at least about 2½ percent diammonium phosphate and wherein the strength of tris-1-aziridinyl phosphine oxide in said solution is at least about 15 percent.

7. A laundry-fast composition according to claim 5, wherein the strength of diammonium phosphate in said solution is at least about 5 percent and the strength of said tris-1-aziridinyl phosphine oxide in said solution is about 15–20 percent.

8. A laundry-fast composition according to claim 5, containing about 5 percent diammonium phosphate and about 18.2 percent tris-1-aziridinyl phosphine oxide.

9. Method for rendering a fibrous organic fabric flame-resistant, comprising impregnating said fabric with an aqueous solution according to claim 5, and curing said impregnated fabric above room temperature until a poly-

mer of tris-1-aziridinyl phosphine oxide is formed in situ thereon.

10. Method according to claim 9, wherein said curing temperature is at least about 250° F.

11. Method for rendering a fibrous organic fabric flame-resistant after repeated launderings, comprising impregnating said fabric with an aqueous solution according to claim 6, and curing said impregnated fabric above room temperature until a polymer of tris-1-aziridinyl phosphine oxide is formed in situ thereon.

12. Method according to claim 11 wherein said curing temperature is at least about 250° F.

13. Method for rendering a cotton fabric flame-resistant after repeated launderings, comprising impregnating said cotton fabric with an aqueous solution of at least about 5 percent diammonium phosphate and at least about 15 percent tris-1-aziridinyl phosphine oxide and curing said impregnated fabric above room temperature until a polymer of tris-1-aziridinyl phosphine oxide is formed in situ thereon.

14. Method according to claim 13, wherein said curing temperature is at least about 250° F.

15. Method according to claim 13, wherein said aqueous solution contains about 5 percent diammonium phosphate and about 18.2 percent tris-1-aziridinyl phosphine oxide.

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