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[54] FLAME RETARDANT FABRIC BLENDS

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[58] **Field of Search** ..... 117/136, 143 A, 138.8 F; 252/8.1

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[57] **ABSTRACT**

Flame retardance-producing compositions for fabric blends comprising thermoplastic fibers, such as polyester fibers, and cellulose fibers, such as cotton fibers, comprising mono (2,3 dibromopropyl) phosphoric acid, a water-soluble cyclic nitrogen-containing polymerizable monomeric compound, and a catalytic amount of tetrakis (hydroxymethyl) phosphonium chloride in aqueous solution, the method of rendering a fibrous organic fabric flame retardant by the application of the flame retardance-producing composition to such a fabric, including drying the fabric, and the flame retardant fibrous organic fabric produced by the method using the flame retardance-producing composition therein.

4 Claims, No Drawings

FLAME RETARDANT FABRIC BLENDS

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

This invention relates to an improved flame retardant composition for fabric or fiber blends comprising thermoplastic fibers and cellulose fibers, more particularly blends of polyester fibers and cellulose fibers, and to the flame-retardant fabrics or fiber blends treated with the improved flame retardant composition. The invention also relates to an improved method for rendering fabric or fiber blends comprising the above-mentioned fibers flame-retardant, and particularly to a method of rendering such fabrics and fiber blends durably flame-retardant with respect to repeated launderings of the treated fabrics or fiber blends under standardized laundering conditions.

It is well-known in the art that mixtures of polyester fibers with cotton fibers, i.e., mixtures of fibers of polyethylene and terephthalate and the like with cotton fibers, are especially difficult to render flame-retardant. Nevertheless, blends of about 50 percent polyester and 50 percent cotton, or blends containing still higher percentages of polyester, are becoming very popular because of the relatively low costs of such fabrics together with the desirable properties which characterize them.

It is, therefore, an object of the present invention to provide an improved flame retardant composition for use in treating fabrics or fiber blends comprising thermoplastic fibers and cellulose fibers to render the same flame-retardant.

Another object of the invention is to provide a method for rendering fabrics or fiber blends comprising thermoplastic fibers and cellulose fibers flame-retardant. A further object is to provide a method for rendering such fabrics or fiber blends durably flame-retardant, i.e., able to retain substantially the degree of flame retardance imparted to them by the treatment with the flame retardant composition after repeated launderings under standardized conditions.

A still further object is to provide flame-retardant fabrics or fiber blends comprising thermoplastic fibers and cellulose fibers, the fabrics or fiber blends being preferably made durably flame-retardant.

Other objects and advantages of the invention will become apparent from the following description of the invention, and the novel features will be particularly pointed out in connection with the appended claims.

The use of methylol-phosphorus polymers as flame retardants for fibrous cellulose materials such as cotton, rayon, linen, ramie, paper, and the like has been known for many years. Also, the use of bromine-containing compounds, especially bromine-containing polymeric materials, as flame retardants for various types of combustible materials, especially for combustible thermoplastic materials, has been known heretofore. We have found that by mixing a bromine-and phosphorous-containing monomeric material, particularly mono (2,3 dibromopropyl) phosphoric acid, with a water-soluble cyclic nitrogen-containing polymerizable monomeric compound, such as trimethylolmelamine or the like, in aqueous solution in the presence of a catalytic amount of tetrakis (hydroxymethyl) phosphonium chloride, a very effective flame retardant

composition is obtained for blends of thermoplasitc fibers with cellulous fibers, such as 50:50 blends of polyethylene terephthalate and cotton. When such an aqueous solution is applied to fabrics prepared from blends of polyesters, such as polyethylene terephthalate, and cotton, in sufficient quantity to result in a dry add-on of at least about 34 percent by weight, and the impregnated fabric is dried, a highly flame-retardant fabric is produced. We have also found that when the water-soluble cyclic nitrogen-containing polymerizable monomeric compound is trimethylolmelamine and the dry add-on is at least about 45 percent by weight, in the case of a 50:50 blend of polyester and cotton fibers in the fabric treated with the flame retardant composition, the dried fabric is very flame-retardant and retains substantially its originally produced flame retardance after at least fifteen launderings under standardized laundering conditions.

The flame retardant treatment is relatively simple and economical, requiring only a one-step application of a single aqueous solution, which may be padded or otherwise conveniently applied to the fabric requiring flame retardance, then drying at a conveniently low temperature, such as from about 100° to 115°C., preferably at about 105°C., to remove the moisture therefrom within a reasonable length of time. The dried, treated fabric may be rinsed in water to remove any unreacted monomers or reaction by-products, and dried again before being tested or used.

Water-soluble cyclic nitrogen-containing polymerizable monomeric compounds suitable for use in the instant invention include triazines, triazones, and cyclic alkylene ureas. Typical examples of suitable triazines include the methylol melamines, such as mono-, di-, and trimethylolmelamine, modified methylol melamines, such as the trimethyl ether or the triethyl ether of trimethylolmelamine. Typical of the triazones suitable for use in the process is dimethylol triazone. Typical examples of suitable cyclic alkylene ureas include dimethylolethylene urea, dimethylolpropylene urea, and dimethyloldihydroxyethylene urea.

Having described the procedure for carrying out the invention in general terms, we will now proceed to illustrate the practice of our invention by giving some examples.

EXAMPLE 1

A 6 oz. per sq. yd. polyester-cotton twill fabric made of 50 percent polyethylene terephthalate fibers and 50 percent cotton fibers was padded with a solution having the following composition so that when dried, the treated fabric had a 45 percent by weight add-on:

Solution Composition

	Parts by Weight
Trimethylolmelamine	100
Mono (2,3 dibromopropyl) phosphoric acid (in 98 percent aqueous solution)	100
Tetrakis (hydroxymethyl) phosphonium chloride (in 80 percent aqueous solution)	24
Water	100

The impregnatd (by padding) fabric was dried at approximately 105°C. for about 20 minutes. The dried fabric was rinsed in water at about 54.5°C. for about 1 hour to remove any unreacted components of the solution and any water-soluble by-products produced along

with the flame-retardant polymer. The treated fabric was dried again after the rinsing and then a portion of the dried fabric was tested for flame retardance in accordance with Method 5903 of Federal Test Method Standard No. 191, dated May 15, 1951. Another portion of the dried fabric was subjected to 15 launderings in accordance with Method 5556 of Federal Test Method Standard No. 191, dated May 15, 1951 and then dried and tested for flame retardance in the same manner as the unlaundered treated fabric. Results of these flame retardance tests are given in Table 1, below. In contrast to these results, the original untreated fabric was completely consumed when subjected to a similar flame retardance test.

TABLE I

Treated Fabric	After-flame (seconds)	After-glow (seconds)	Char length (inches)
Initial	0	1	4.3
After 15 launderings	0	2	5.0

It is apparent that the treated fabric was quite flame retardant and that after 15 launderings under standardized conditions its flame retardance was still almost as great as initially following treatment with the flame retardant solution.

EXAMPLE 2

A polyester-cotton twill fabric of the same weight and composition as that of Example 1 was padded with a solution having the following composition so that when dried, the treated fabric had a 34 percent by weight add-on:

Solution Composition

	Parts by Weight
Dimethyloldihydroxyethylene urea (in 47 percent aqueous solution)	200
Mono (2,3 dibromopropyl) phosphoric acid (in 98 percent aqueous solution)	100
Tetrakis (hydroxymethyl) phosphonium chloride (in 80 percent aqueous solution)	24

The impregnated (by padding) fabric was dried at approximately 105°C. for about 20 minutes, then cured at approximately 160°C. for about 5 minutes. The cured fabric was rinsed in warm water for about 5 minutes to remove any unreacted components of the solution and any by-products produced along with the flame-retardant polymer. The treated fabric was dried again and a portion of the dried fabric was tested for flame retardance in the same manner as the fabric in Example 1. It exhibited an afterflame time of zero seconds, an after glow time of zero seconds, and a char length of 5.7 inches, which represents a satisfactory degree of flame retardance for many uses. The untreated fabric was completely consumed as in Example 1.

While the foregoing examples are in terms of application of the flame retardant compositions of the invention to 50:50 blend polyester-cotton fabrics, it is to be understood that other blends of polyester fibers, or other thermoplastic fibers, and cellulose fibers may be rendered flame-retardant by treatment with aqueous solutions of mono (2,3 dibromopropyl) phosphoric acid and a water-soluble cyclic nitrogen-containing polymerizable monomeric compound together with a catalytic amount of tetrakis (hydroxymethyl) phosphonium chloride and that in many cases simply drying the impregnated fabric treated with the flame retardant solution is adequate for producing the necessary amount of polymerization. The method of the invention is particularly effective for fabrics or fiber blends having a high percentage of the thermoplastic fibers therein. With some of the polymerizable monomeric compounds, however, some curing at an elevated temperature, such as about 150°–177°C., the conventional curing range used in the textile finishing art, may be desirable, especially with other cyclic nitrogen-containing monomeric compounds than the methylolmelamines.

We are, of course, aware that tetrakis (hydroxymethyl) phosphonium chloride has been used heretofore in conjunction with trimethylolmelamine and other similar cyclic nitrogen-containing polymerizable monomers in the production of flame retardance in fabrics, particularly cotton, rayon, ramie, jute, and even wool. However, in such cases rather large proportions of the tetrakis (hydroxymethyl) phosphonium chloride have been required to produce a reasonably high degree of flame retardance. The important contribution we have made to the flame retardance of fabric blends containing substantial proportions of the thermoplastic fibers with our invention is in taking advantage of the ability of tetrakis (hydroxymethyl) phosphonium chloride in relatively low proportions to catalyze the copolymerization of a known monomeric flame retardant for polyester fibers and other thermoplastic fibers, namely mono (2,3 dibromopropyl) phosphoric acid, with cyclic nitrogen-containing polymerizable monomeric compounds, such as trimethylolmelamine, in a single aqueous solution, such that in at least some instances drying only at relatively low temperatures is sufficient to effect the polymerization of the monomers to flame retardance-imparting polymers, and further that in at least some instances the polymers formed in situ in the fabric blends are retained therein through at least 15 launderings under standardized conditions in sufficient quantities to maintain sufficient flame retardance in the fabric blends to satisfy military requirements as defined in military specifications. Most flame retardant treatments which have imparted laundering resistance heretofore have necessitated high temperature curing treatments in contrast to the preferred process and the preferred composition of the present invention.

The tetrakis (hydroxymethyl) phosphonium chloride employed as catalyst in the method of the invention will preferably be present in the flame retardant composition to the extent of at least about 7.4 percent. A higher concentration thereof may be employed; however, there may be danger of some damage to the fabric, particularly the cellulose fibers, if the catalyst is present in much greater concentration than about 7.4 percent, which is adequate in most cases.

The flame retardant composition and process of the present invention are useful for imparting durable flame retardance to blends of thermoplastic fibers with cellulose fibers, and especially blends of polyester fibers, such as polyethylene terephthalate fibers, with cotton fibers, which is fast becoming an extremely popular blend because of the trend toward lower costs of production of the polyester fibers and the ability of

such fibers to modify and complement the characteristics of cotton fibers so that such blends possess good strength and durability while retaining sufficient moisture absorptivity to make them reasonably comfortable for wearing apparel. Crease retention is another desirable characteristic of many of these blends. Now, as a result of the present invention, such blends may be easily and economically rendered flame-retardant and such flame retardance may be made durable to repeated launderings without unduly increasing the costs of the fabrics or clothing items produced therefrom.

We wish it to be understood that we do not desire to be limited to the exact details described for obvious modifications will occur to a person skilled in the art.

We claim:

1. A flame-retardant cotton-polyester blend fabric comprising about 50 percent cotton fibers in the fibrous portion of said fabric, said fabric having deposited therein a polymeric reaction product produced by drying in said fabric an aqueous solution impregnated in said fabric, said aqueous solution comprising mono (2,3 dibromopropyl) phosphoric acid, a water-soluble cyclic nitrogen-containing polymerizable monomeric compound, and tetrakis (hydroxymethyl) phosphonium chloride, said tetrakis (hydroxymethyl) phosphonium chloride being present in said aqueous solution in an amount effective to catalyze polymerization of said mono (2,3 dibromopropyl) phosphoric acid with said water-soluble cyclic nitrogen-containing polymerizable monomeric compound, said polymeric reaction product being deposited in said fabric in an amount effective to make said fabric flame retardant and to cause said fabric to retain flame retardancy after 15 launderings under standardized conditions.

2. A flame-retardant cotton-polyester blend fabric according to claim 1, wherein said polymeric reaction product is deposited in said fabric in an amount from about 34 percent to about 45 percent of the weight of said fabric.

3. A flame-retardant cotton-polyester blend fabric according to claim 1, wherein said water-soluble cyclic nitrogen-containing polymerizable monomeric compound is selected from the group consisting of triazines, triazones, and cyclic alkylene ureas.

4. A flame-retardant cotton-polyester blend fabric according to claim 3, wherein said water-soluble cyclic nitrogen-containing polymerizable monomeric compound is trimethylolmelamine.

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