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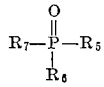
PROCESS FOR PREPARING POLYESTER BLEND FABRICS WITH DURABLE FLAME RESISTANCE

Stancil Driftwood Powell, Charlotte, N.C., assignor to Celanese Corporation, New York, N.Y.

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U.S. Cl. 117—76 T 5 Claims

ABSTRACT OF THE DISCLOSURE

There is provided a process for imparting flame retardancy to a fabric comprised of at least 30 percent of polyester fiber, at least 5 percent of cellulose acetate fiber, and at least 15 percent of a cellulosic fiber. In said process of the fabric is passed through a bath comprised of a flame retardant amount of tetrakishydroxymethylphosphonium chloride. Thereafter the fabric is cured at a temperature of from about 225 to about 400 degrees Fahrenheit for from about 15 seconds to about 10 minutes. To impart flame retardancy to the polyester portion of said fabric the fabric is passed through a solution or emulsion comprised of a flame retardant amount of a compound of the formula



wherein R_5, R_6, and R_7 are selected from the group consisting of hydroxy, chlorine, fluorine, bromine, iodine, haloalkyl of 1 to about 18 carbon atoms, and halohydroxyalkyl of 1 to about 18 carbon atoms, provided that at least one of R_5, R_6, and R_7 is haloalkyl or halohydroxyalkyl. The use of cellulose acetate as one of the components of the aforementioned fabric allows one to obtain more durable flame retardance and better fabric aesthetics with reduced quantities of flame retardant.

This invention relates to a process for preparing a unique flame resistant polyester blend fabric.

Blends of polyester and cellulose are more flammable than either of the two components. One means of rendering these blends flame retardant is to treat them with flame retardants specific for each component; thus, e.g., one may treat these blends with tetrakishydroxymethylphosphonium chloride (for the cellulosic component) and 2,3 dibromopropyl phosphate (for the polyester component).

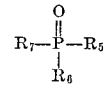
The prior art "dual flame retardant" systems employed suffer from the disadvantage that the amount of flame retardant one need apply to make the cellulosic component durably flame-resistant is relatively large, and the use of such an amount of flame retardant ruins the aesthethics of the fabric, giving one a fabric which is too "boardy."

It is thus an object of this invention to provide a process for imparting durable flame retardance to blends of polyester and cellulose wherein the aesthetics of the fabric are not ruined. In accordance with this invention, there is provided a process for imparting flame retardancy to a fabric comprised of from about 30 to about 80 percent (by weight of fabric) of polyester fiber and from about 5 to about 50 percent (by weight of fabric) of cellulose acetate fiber, the remainder of said fabric being comprised of at least 15 percent (by weight of fabric) of a cellulosic fiber, comprising the steps of:

(1) Passing said fabric through a bath comprised of a flame-retardant amount of tetrakishydroxymethylphosphonium chloride;

(2) Curing said treated fabric by subjecting it to a temperature of from about 225 to about 400 degrees Fahrenheit for from about 15 seconds to about 10 minutes; and

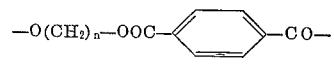
(3). Passing said fabric through a solution or emulsion comprised of a flame-retardant amount of a compound of the formula



wherein R_5, R_6, and R_7 are selected from the group consisting of hydroxy, chlorine, fluorine, bromine, iodine, haloalkyl of 1 to about 18 carbon atoms, and halohydroxyalkyl of 1 to about 18 carbon atoms, provided that at least one of R_5, R_6, and R_7 is haloalkyl or halohydroxyalkyl.

It has been discovered that, unexpectedly, the use of the cellulose acetate in the aforementioned fabric allows one to obtain more durable flame retardance with reduced quantities of tetrakishydroxymethylphosphonium chloride with the process of this invention.

The fabric treated in applicant's process is comprised of from about 30 to about 80 percent (by weight of fabric) of polyester fiber, although it is preferred that said fabric be comprised of from about 50 to about 70 percent (by weight of fabric) of polyester fiber. The term "polyester fiber," as used herein, refers to a fiber in which the fiber-forming substance is any long chain synthetic polymer composed of at least 85 percent (by weight) of an ester of a dihydric alcohol and terephthalic acid. Thus, e.g., a fabric comprised of the ester of 1,4-cyclohexanedimethanol and terephthalic acid works well in the process of this invention. The preferred polyester fiber is made from an ester of a diol of the formula $HO(CH_2)_n OH$ (wherein n is from 2 to 10) and terephthalic acid and is comprised of recurring structural units of the formula



The most preferred polyester fiber is poly(ethylene terephthalate) fiber.

The fabric treated in applicant's process is also comprised of from about 5 to about 50 percent (by weight of fabric) of cellulose acetate fiber, although it is preferred that it be comprised of from about 20 to about 40 percent (by weight of fabric) of cellulose acetate fiber. The term cellulose acetate fiber, as used herein, refers to any fiber in which the fiber-forming substance is cellulose acetate (and includes, e.g., cellulose triacetate wherein at least 92 percent of the hydroxyl groups are acetylated). The cellulose acetate may be partially saponified or unsaponified, although it is preferred that it be unsaponified. When saponified cellulose triacetate fiber is used in the process of this process, it is preferred that only the surface of said fiber be saponified to an acetyl value of about 50. The most preferred cellulose acetate fiber is cellulose triacetate fiber.

The fabric treated in applicant's process is also comprised of at least 15 percent (by weight of fabric) of

cellulosic fiber. The term cellulosic fiber, as used herein, refers to, e.g., cotton, flax, rayon, viscose, high wet modulus rayon, and the like. The most preferred cellulosic fibers are selected from the group consisting of cotton fiber and rayon fiber.

In the process of this invention said fabric is passed through a bath comprised of a flame retardant amount of tetrakishydroxymethylphosphonium chloride.

A solution or dispersion of a compound of the formula



wherein R_5, R_6, and R_7 are selected from the group consisting of hydroxy, chlorine, fluorine, bromine, iodine, haloalkyl of 1 to about 18 carbon atoms, and halohydroxyalkyl of 1 to about 18 carbon atoms, provided that at least one of R_5, R_6, and R_7 is haloalkyl or halohydroxyalkyl is used to impart flame retardancy to the polyester component of said blend. The fabric is passed through a solution or emulsion comprised of a flame retardant amount of said compound either before or after it is passed through said bath; it is preferred to first pass the fabric through said bath, cure it, and then pass it through said solution or emulsion.

It is preferred that R_5, R_6, and R_7 be selected from the group consisting of chloroalkyl and bromoalkyl of 1 to about 8 carbon atoms. It is even more preferred that said R substituents be dibromopropyl substituents, most preferably 2,3-dibromopropyl substituents.

Thus the most preferred flame retardants used in the process of this invention are tetrakishydroxymethylphosphonium chloride (for the cellulosic component) and 2,3-dibromopropyl phosphate (for the polyester component). For the sake of convenience, the process of this invention will be described with regard to these flame retardants, it being understood that the process parameters being described are equally applicable to the other flame retardants which may be used in the process of this invention.

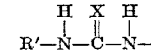
In the process of this invention said fabric is passed through a bath comprised of from about 50 to about 400 grams tetrakishydroxymethylphosphonium chloride where-in said bath has a pH of from about 6 to about 9. Both said tetrakishydroxymethylphosphonium chloride and 2,3-dibromopropyl phosphate may be present in the same bath, although it is preferred to have them in separate baths; when they are in the same bath the 2,3-dibromopropyl phosphate may be emulsified. In one preferred embodiment of applicant's invention wherein only one bath is used, the tetrakishydroxymethylphosphonium chloride may be neutralized with caustic soda to a pH of from about 6 to about 9 (and preferably from about 7.0 to about 7.6), urea may be dissolved in the bath, emulsifier may be added, and with high speed agitation a 50 to 60 percent solution of 2,3 dibromopropyl phosphate in xylene is added. Resins may be added to the bath either prior to or subsequent to the addition of the 2,3 dibromopropyl phosphate. The aforementioned merely exemplifies one of the many embodiments of applicant's invention and is not to be deemed limitative thereof.

It is preferred that said bath comprised of said tetrakishydroxymethylphosphonium chloride ("THPC") be comprised of from about 100 to about 300 grams per liter of bath of THPC and that the pH of said bath be from about 7 to about 8. It is most preferred that said bath be comprised of from about 150 to about 250 grams per liter of bath of THPC.

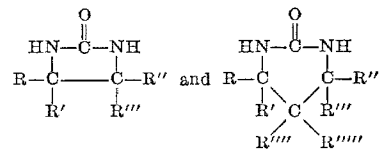
Said bath comprised of THPC may be maintained at the desired pH by adding to the bath, e.g., enough alkali metal hydroxide to obtain the desired pH. The preferred alkali metal hydroxide is sodium hydroxide, and generally from about 5 to about 30 grams thereof per liter of bath may be used to give one the desired pH. Any other effective neutralizing agent may also be used.

Said bath comprised of THPC may also be comprised of from about 25 to about 300 grams per liter of bath of a cross linking resin. The cross-linking resins used by the art may comprise said bath. The preferred cross-linking resins include, e.g., resins comprised of dimethylol urea, trimethylol melamine, dimethylol ethylene urea, dimethylol propylene urea, dimethylol dihydroxy ethylene urea, dimethylol ethyl triazine, and dimethylol monocarbonate. Other suitable cross-linking resins include, e.g., dimethylolhydroxyethyl triazone, dimethylol uron, dimethyoxydimethylol uron, and the like.

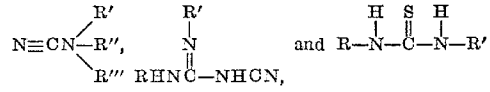
Said bath comprised of THPC may also be comprised of from about 15 to about 50 grams per liter of bath of a nitrogen compound which forms an insoluble polymer with THPC when subjected to the curing step of the process of this invention. Thus, e.g., a compound of the formula



(wherein said R substituents are independently selected from the group consisting of hydrogen, hydroxy, alkyl of from 1 to about 8 carbon atoms, aryl (such as, e.g., phenyl), and halogen selected from the group consisting of fluorine, chlorine, bromine, and iodine; and wherein X is selected from the group consisting of oxygen and sulfur) work well in the process of this invention. The preferred compounds from this group are selected from the group consisting of urea and thiourea, and the most preferred compound from this group is urea. Thus, e.g., compounds of the formulae



(wherein said R substituents are hereinbefore described) work well in the process of this invention; the most preferred compounds from this group are cyclic ethylene urea and cyclic propylene urea. Thus, e.g., compounds of the formulae RN=C=NR',



(wherein said R substituents are as hereinbefore described) work well in the process of this invention; the preferred compounds from this group are cyanimide, dicyandiamide, and guanidine. Thus, e.g., alkyl carbamates (such as ethyl, propyl, and octyl carbamate) work well in the process of this invention. It is to be understood that when applicant refers to "said R substituents" he is referring to the substituents denoted as R, R', R'', R''', R'''', and R'''''.

When said nitrogen compound is used in said bath it is preferred that it be urea.

The bath comprised of THPC may also be comprised of from about 0.1 to about 10 grams per liter of bath of a wetting agent such as, e.g., a wetting agent, comprised of, e.g., alkyl aryl ethoxylated phenol (e.g., a dodecyl benzene sulfonic acid salts).

After the fabric of this invention has been passed through the bath comprised of THPC, it may be dried at a temperature of from about 200 to about 280 degrees Fahrenheit.

After the fabric has been passed through the THPC bath, it is cured at a temperature of from about 225 to

about 400 degrees Fahrenheit for from about 15 seconds to about 10 minutes, whereby the THPC is fixed onto said fabric. It is preferred to use a curing temperature of from about 300 to about 400 degrees Fahrenheit. It is more preferred to cure the fabric at a temperature of from about 325 to about 375 degrees Fahrenheit for from about 1 to about 4 minutes. It is most preferred to cure the fabric at a temperature of 330 degrees Fahrenheit for about 3 minutes.

After the fabric has been cured, it is passed into a solution or emulsion of 2,3-dibromopropyl phosphate which is comprised of from about 2 to about 25 percent (by weight of solution or emulsion) of 2,3-dibromopropyl phosphate. Suitable solvents which may be employed to make said solution include chlorinated hydrocarbons such as perchloroethylene, 1,1,1-trichloroethane, methylene chloride, carbon tetrachloride, chloroform, and the like. Suitable emulsifying agents may be used to form said emulsion of 2,3-dibromopropyl phosphate include, e.g., emulsifiers comprised of polyglycol ethers (such as e.g., "Tanapon X–70"). In general any of the emulsifying agents known to the art which emulsify 2,3-dibromopropyl phosphate may be used in the process of this invention.

It is preferred that the bath ocomprised of 2,3-dibromopropyl phosphate have from about 5 to about 20 percent (by weight of emulsion or solution) of 2,3-dibromopropyl phosphate, and it is even more preferred that it be comprised of about 15 percent of said 2,3 dibromopropyl phosphate.

After the fabric has been passed through the second bath, it may be washed.

In order to illustrate some of the preferred embodiments of his invention, applicant presents the following examples which are not to be deemed limitative of his invention. Unless otherwise stated, all parts are by weight and all temperatures are in degrees Fahrenheit.

EXAMPLES

Example 1

A cellulose triacetate/polyester/cotton broadcloth fabric comprised of 33.3 percent of each of the aforementioned components was padded with a bath comprised of 100 grams per liter of 80 percent THPC, 50 grams per liter of 20 percent caustic, 17 grams per liter of urea and 100 grams/liter of a melamine-formaldehyde resin; this solution was padded onto the fabric to a wet pick-up of from about 70 to about 100 percent. Thereafter the fabric was dried, cured at a temperature of 330 degrees Fahrenheit for 3 minutes, and padded with a solution comprised of 10 percent (by weight of solution) of 2,3-dibromopropyl phosphate. The fabric was then dried 2 mintues at a temperature of 330 degrees Fahrenheit, padded with a bath comprised of 60 grams per liter of ammonium sulfate, 36 grams per liter of soda ash, and 30 grams per liter of sodium bicarbonate, and then washed for 20 minutes at 140 degrees Fahrenheit in a solution comprised of 1 gram per liter of sodium perborate and 1 gram per liter of soda ash. Then the fabric was dried.

The fabric was then washed over 100 times with the "home cycle" at a temperature of from about 140 to about 150 degrees Fahrenheit; 50 cc. of "Tide" detergent were used for each washing.

The fabric was then tested for flammability in accordance with AATCC Vertical Flame Test 34–1966 wherein a flame was applied to the fabric for 12 seconds and then removed and the extent of char and the amount of afterflame that occurred were observed. After being subjected to this test the fabric had a 5.9 inch char and no afterflame.

Example 2

A cellulose triacetate/polyester/high wet modulus rayon basket weave fabric was treated and tested in accordance with Example 1. The fabric had a 3.7 inch char and no afterflame.

Example 3

60/20/20 and 50/25/25 blends of each of the cellulose triacetate/polyester/cellulosic fabrics of Examples 1 and 2 were treated and tested in accordance with the procedure of Example 1; they exhibited comparably good inflammability properties.

Example 4

In accordance with the procedure described in Example 1, a 50/50 polyester/cotton broadcloth fabric was washed only 20 times and then tested. The fabric had a 10 inch char and 20 seconds afterflame.

Although the above examples and descriptions of the invention have been very specifically illustrated, many other modifications will suggest themselves to those skilled in the art upon a reading of this disclosure; these are intended to be comprehended within the scope of this invention.

What is claimed is:

1. A process for imparting durable flame retardancy to a fabric comprised of from about 30 to about 80 percent, by weight of fabric, of polyester fiber and from about 5 to about 50 percent, by weight of fabric, of cellulose acetate fiber, the remainder of said fabric being comprised of at least 15 percent, by weight of fabric, of a cellulosic fiber, comprising the steps of sequentially:
   (a) passing said fabric through a bath comprised of from about 50 to about 400 grams per liter of bath of tetrakishydroxymethylphosphonium chloride, wherein the pH of said bath is from about 6 to about 9;
   (b) curing said treated fabric by subjecting it to a temperature of from about 225 to about 400 degrees Fahrenheit for from about 15 seconds to about 10 minutes; and
   (c) passing said fabric through a solution or emulsion comprised of from about 2 to about 25 percent, by weight of said solution or emulsion, of 2,3-dibromopropyl phosphate.

2. The process of claim 1, wherein:
   (a) said cellulose acetate fiber is cellulose triacetate fiber, and said polyester fiber is poly(ethylene terephthalate) fiber;
   (b) said fabric is passed through a bath comprised of from about 100 to about 300 grams per liter of tetrakishydroxymethylphosphonium chloride, dried at a temperature of from about 200 to about 280 degrees Fahrenheit, and then cured at a temperature of from about 300 to about 400 degrees Fahrenheit for from about 1 to about 4 minutes; and
   (c) thereafter said fabric is passed through a solution or emulsion comprised of from about 5 to about 20 percent (by weight of said solution or emulsion) of 2,3-dibromopropyl phosphate.

3. The process of claim 2, wherein said bath comprised of tetrakishydroxymethylphosphonium chloride is also comprised of from about 25 to about 300 grams per liter of bath of a cross-linking resin selected from the group consisting of dimethylol urea, trimethylol melamine, dimethylol ethylene urea, dimethylol propylene urea, dimethylol dihydroxy ethylene urea, dimethylol ethyl triazine, dimethylol monocarbamate, dimethylolhydroxyethyl triazone, dimethylol uron, and dimethoxydimethylol uron.

4. The process of claim 3, wherein:
   (a) said cellulosic fiber is selected from the group consisting of cotton and rayon;
   (b) said bath comprised of tetrakishydroxymethylphosphonium chloride additionally contains from about 15 to about 50 grams per liter of urea; and
   (c said treated fabric is cured at a temperature of from about 325 to about 375 degrees Fahrenheit.

5. The process of claim 4 wherein said cellulosic fiber is cotton; said treated fabric is cured by subjecting it to a temperature of about 330 degrees Fahrenheit for about

3 minutes; and said solution or emulsion is comprised of about 15 percent (by weight) of 2,3-dibromopropyl phosphate.

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WILLIAM D. MARTIN, Primary Examiner

H. J. GWINNELL, Assistant Examiner

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