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3,193,602

PROCESS FOR THE PRODUCTION OF FLAME RETARDED ACRYLIC FIBERS

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7 Claims. (Cl. 264-182)

This invention is concerned with improving textile properties of wet spun flame retarded synthetic linear fibers. More specifically, this invention is concerned with a method of improving the textile properties of wet spun flame retarded fiber, containing as the major part of the fiber, acrylonitrile.

Many of the fibers composed primarily of acrylonitrile when tufted into carpets, produce a carpet which has excellent textile properties, such as abrasion resistance, a very good resilience, resistance to matting and soilage and other desirable properties. However, there is a problem here of a propensity for these fibers to support a flame. In the past, when various flame resisting or flame retarding agencies were incorporated within these fibers, the textile properties, such as those listed above, were harmed and carpets produced from these fibers were not of commercial acceptability. If, for example polyvinylchloride is used as a flame retarding agent and incorporated with an acrylic fiber, after 32,000 traffic, the rug is severely matted, a great deal of its resiliency is lost, and a large number of fibrils have broken loose from the fibers, whereas a similar fiber not incorporating polyvinylchloride produced a carpet which was still in good shape after 64,000 traffic.

An object of this invention is to provide a method for producing flame retarded fibers which retain good textile properties.

Another object of this invention is to provide a method for the production of flame retarded acrylic fibers which possess excellent abrasion characteristics.

Another object of this invention is to provide a method for the production of flame retarded acrylic fibers which have excellent resiliency characteristics.

Another object of this invention is to provide a method for the production of flame retarded acrylic fibers which will resist matting.

Another object of this invention is to provide a method for the production of flame retarded acrylic fibers which possess excellent soilage resistance.

Other objects and advantages of this invention will become apparent from the hereinafter detailed description.

The objects of this invention are generally accomplished by spinning the fibers into a spin bath of high solvent and low temperature when compared to the conventional wet spin bath. In addition, the jet stretch is lower than in the conventional wet spin method for acrylonitrile based fibers.

More specifically, the polymers are spun into a wet spin bath in which the solvent, based upon the total weight of the spin bath, may vary from 50 percent to 75 percent, with the preferred depending upon the specific polymer composition as described hereinafter. This solvent in the spin bath is normally the same solvent in which the polymer is dissolved prior to spinning. The water portion of the spin bath will comprise the remainder. In addition, this spin bath is maintained within a temperature range of from 0° C. to 36° C. with the preferred temperature being 26° C. Under the normal wet spinning method for acrylonitrile based fibers, there is a jet stretch ratio of 0.7 to 2.0; however in the process of the present invention this jet stretch ratio may vary from 0.1 to 0.6 with the preferred jet stretch ratio

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being 0.3. This jet stretch is the ratio of the linear rate of withdrawal of fibers from the spin bath to the linear rate of extrusion of dope into the spin bath. The method of the invention is applicable primarily to the spinning of heavy denier fibers varying from 10 to 40 denier per filament.

While this application has been generally directed to synthetic fibers, it is especially useful in the wet spinning of fibers composed of acrylic polymers and more especially when these fibers have incorporated within them flame retarding agents such as halogenated long chain fatty acid esters, aliphatic phosphates, halogenated aliphatic phosphates, polymeric compositions of vinyl halide and vinylidene halides and other organic retarding agents known to the art. Among those polymeric fibers upon which the method of this invention is especially applicable are copolymers, terpolymers and blends of polymers, for example; a copolymer of 85 percent acrylonitrile and 15 percent vinylchloride; a terpolymer of 85 percent acrylonitrile, 10 percent vinylidene chloride and 5 percent vinyl acetate; and a fiber composed of 85.3 percent of a blend of two copolymers, the first copolymer being 88 percent of 94 percent acrylonitrile and 6 percent vinyl acetate and the second copolymer being 12 percent of 50 percent acrylonitrile and 50 percent methyl vinylpyridine with 12.8 percent polyvinylchloride and 1.9 percent antimony trioxide added as flame retardant agents. While the method of this present invention is especially applicable to the above identified fibers or polymers, it is generally applicable to any of the fibers which contain a flame retarding agent, generally listed hereinbefore, and a major part of the fiber being acrylonitrile.

The following examples are cited to illustrate the invention. They are not intended to limit it in any way. Unless otherwise noted, percentages as expressed in the examples indicate percent by weight.

Example 1

A fiber composition composed of 85.3 percent of a blend of two copolymers, the first copolymer being 88 percent of 94 percent acrylonitrile and 6 percent vinyl acetate and the second copolymer being 12 percent of 50 percent acrylonitrile and 50 percent methyl vinylpyridine with 12.8 percent polyvinylchloride and 1.9 percent of antimony trioxide, was dissolved in a dimethylacetamide solvent, representing a 19 percent solid dope. This composition was spun into a coagulating bath comprising 66 percent of a dimethylacetamide solvent and 34 percent water at 20° C. and a jet stretch of 0.4. These conditions varied from the normal coagulating conditions of 30 to 60 percent of a dimethylacetamide solvent to 60 to 35 percent water at 40° C. to 60° C. which are fully satisfactory for spinning a similar fiber forming composition not including the flame retardant polyvinylchloride. A microscopic examination of the uncollapsed fiber produced under the new conditions, showed that the high solvent-low temperature fibers produced fewer voids than the same fiber composition spun under the normal coagulating conditions. Rugs were subsequently tufted of 15 denier per filament of both of these fibers and were compared as to their textile properties. The resiliency, abrasion resistance and resistance to matting and soilage of the fibers produced by the method of this invention were very superior to the fibers produced in the normal coagulating bath.

Example 2

A copolymer of 85 percent acrylonitrile and 15 percent vinylidene chloride was spun to a fiber of 10 denier per filament at a jet stretch of 0.3 in a spin bath of 67 percent of dimethylacetamide solvent held at a tempera-

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ture of 20° C. Water was present in the amount of 33 percent. The fibers produced were essentially free of voids, and when they were tufted into carpets, the resiliency, abrasion resistance and resistance to matting and soilage were very superior to similar fibers produced in the normal coagulating bath.

Example 3

A terpolymer of 85 percent acrylonitrile, 10 percent vinylidene chloride and 5 percent vinyl acetate was spun into a coagulating bath composed of 50 percent, plus or minus 2 percent, of a dimethylacetamide solvent, temperature at a range of 18° C. to 26° C. and water in the amount of 50 percent, plus or minus 2 percent, with a jet stretch of 0.26. The 18 denier per filament fibers produced were essentially free of voids and when tufted into carpets, gave excellent resiliency, abrasion resistance and resistance to matting and soilage when compared to carpets produced from fibers coagulated in a normal spin bath as defined above in Example 1.

It is thus apparent that a low bath temperature is of importance in the method of this invention and while certain specific bath concentrations should be maintained for a given polymer composition, the coagulating bath concentration values must be adjusted slightly for different polymer compositions to compensate for different degrees of solubility of various polymer compositions in the spin bath.

Example 4

A fiber blend of 87 percent of a copolymer of 94 percent acrylonitrile and 6 percent vinyl acetate and 13 percent tris (beta-chloroethyl) phosphate was spun into a wet spin bath composed of 70 percent of a dimethylacetamide solvent, 30 percent water, at a temperature reading of 0° C. and a jet stretch of 0.32. The fibers produced were essentially free of voids and when they were tufted into carpets, the resiliency, abrasion resistance and resistance to matting and soilage was superior to the similar fibers produced in the normal coagulating bath.

Example 5

A blend of 89 percent of a copolymer, the first copolymer being 88 percent of 94 percent acrylonitrile and 6 percent vinyl acetate and the second copolymer being 12 percent of 50 percent acrylonitrile and 50 percent methyl vinylpyridine with 11 percent of tri(2-ethylhexyl) phosphate was spun into a coagulating bath composed of 71 percent of a dimethylacetamide solvent, 29 percent water, at a temperature of 23° C. with a jet stretch of 0.34. The fibers produced were essentially free of voids, and when they were tufted into carpets, the resiliency, abrasion resistance and resistance to matting and soilage was superior to similar fibers produced in the normal coagulating bath.

Example 6

A blend of 90 percent of a copolymer of 94 percent acrylonitrile and 6 percent vinyl acetate with 10 percent chlorinated methyl stearate (37.5 percent chlorine) was spun into a spin bath composed of 64 percent of a dimethylacetamide solvent, 36 percent water, at a temperature of 30° C. and a jet stretch of 0.34. The fibers produced were essentially free of voids and when they were tufted into carpets, the resiliency, abrasion resistance and resistance to matting and soilage was very superior to similar fibers produced in the normal coagulating bath.

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Thus with the wet spinning method of this invention, flame retarded acrylonitrile based fibers are produced in which the addition of the flame retarding agent does not result in considerable degraded wear and textile properties. In fact the spinning performance is improved and the textile wear characteristics of carpets made from the fibers produced by the method of this invention are considerably superior to standard wet spun fibers.

It is understood that changes and variations may be made in the present invention by one skilled in the art without departing from the spirit and scope thereof as defined in the appended claims.

We claim:

1. A method for the production for a flame retarded acrylic fiber comprising at least 85 percent acrylonitrile and up to 15 percent of another mono-olefinic monomer copolymerizable therewith to which has been added a flame retarding agent selecting from the group consisting of halogenated long chain fatty acid esters, aliphatic phosphates, halogenated aliphatic phosphates, polyvinyl halides and vinylidene halides, said fibers being between 10 and 40 denier per filament, comprising spinning a solution of said fiber into a spin bath being comprised of at least 60 percent of a dimethylacetamide solvent with water comprising the remainder at a temperature of below 36° C. with a jet stretch of below 0.6.
2. The method of claim 1 in which the flame retardant is polyvinylchloride.
3. The method of claim 1 in which the flame retardant is tris(beta chloroethyl) phosphate.
4. The method of claim 1 in which the flame retardant is tri(2-ethylhexyl) phosphate.
5. The method of claim 1 in which the flame retardant is chlorinated methyl stearate, 37.5 percent chlorine.
6. A method for the production of a flame retardant acrylic fiber comprising at least 85 percent acrylonitrile, 10 percent vinylidene chloride and 5 percent vinyl acetate comprising spinning a solution of said terpolymer into a spin bath composed of 50 percent organic solvent, said solvent being chemically identical to the solvent of said solution, 50 percent water at a temperature of 18° C. to 26° C. with a jet stretch of 0.2 to 0.4.
7. A method for the production of a flame retardant acrylic fiber being comprised of 85 percent of a blend of two copolymers, the first copolymer being 88 percent of 94 percent acrylonitrile and 6 percent vinyl acetate and the second copolymer being 12 percent of 50 percent acrylonitrile and 50 percent methyl vinylpyridine with 13 percent polyvinylchloride and 2 percent antimony trioxide, comprising spinning a solution of said blend into a spin bath composed of 63 percent to 68 percent organic solvent, said solvent being chemically identical to the solvent of said solution, water comprising the remainder, at 20° C. to 30° C. with a jet stretch of 0.2 to 0.4.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

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It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 3, line 35, for "0° C." read -- 20° C. --.

Signed and sealed this 18th day of January 1966.

(SEAL)

Attest:

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