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2,953,480

PROCESS FOR PRODUCING FIRE-RETARDANT NYLON FABRIC

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The present invention relates to a fire-retardant treatment for flammable hydrophobic synthetic textile materials and the resulting product.

A wide variety of substances have been applied to textile fabrics woven from natural fibers and viscose rayon for the purpose of fireproofing the fabrics with varying degrees of success. Phosphates, borates, metal oxides, and ammonium salts including the bromide are among the great number of compounds which have been suggested. Practically all of the fire-retardant agents have been found to be either ineffective or otherwise undesirable for use on the synthetic hydrophobic textiles. These materials pose special problems due to their unusual characteristics as, for instance, their behavior in a flame which differs considerably from that of the natural fibers. A number of the synthetic fibers melt before burning and then drip or fall off the fabric as molten flaming pellets. This phenomenon not only spreads the fire but also resulted in very severe burns to persons wearing apparel made from such textile fibers.

An object of the invention is to provide an improved fire-retardant treatment for fibrous materials of nitrogen containing hydrophobic synthetic fibers.

Another object of the invention is to provide improved flame-resistant textile materials of nitrogen containing hydrophobic synthetic fibers.

A further object of the invention is to provide improved flame-resistant stiffened textile materials containing nylon fibers.

Other objects of the invention will be apparent to those skilled in the art from the detailed description hereinbelow.

The present invention is concerned with a process of applying an aqueous solution of ammonium bromide and an aminotriazine-aldehyde condensate to a flammable material containing synthetic hydrophobic fibers and curing the condensate to the water-soluble state as well as the resulting fire-resistant product.

The present treatment is highly effective with flammable, hydrophobic synthetic fibrous materials. Hydrophobic fibers absorb little or no water; hence viscose and cupro-ammonium rayon fibers are outside of the scope of this invention even if they are considered to be synthetic materials. However, it is operative with cellulose acetate and other polyesters including polyethylene glycol terephthalate, whereas prior art compositions do not provide satisfactory flame-resistance. The invention is particularly adapted for the treatment of nitrogen-containing synthetics such as the linear polyamides known as nylon and acrylic fibers containing homopolymers of acrylonitrile or copolymers with suitable ethylenic compounds such as methyl acrylate, 2-methyl 5-vinyl pyridine, other vinyl pyridines, vinyl acetate, vinyl chloride, etc. All of these flammable hydrophobic synthetic fibers appear to fall into the same group in respect to the problem of obtaining flame-resistance.

Tests on cellulose acetate, nylon and acrylic fabrics have indicated very unsatisfactory fireproofing not only

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with the commercially-used fire-retardants such as the monomethylol dicyandiamide-phosphoric acid mixture, antimony trioxide and polyvinyl chloride latex, mono- and diammonium phosphates and ammonium sulfamate, but also prior art agents including ammonium chloride, phosphoric acid, mixed borax and boric acid, chlorides of calcium, magnesium, zinc, copper, chromium, cobalt and nickel, ammonium sulfate, etc. Based on a lesser but still substantial amount of data, it is apparent that admixture with a melamine resin such as methylated trimethylol melamine will not procure adequate flame retarding qualities on the synthetic fibrous materials. In addition, some of the known fireproofing treatments produce other undesirable results as exemplified by the discoloration of polyacrylonitrile fabrics by alkaline substances. From this it is apparent that the flameproofing of synthetic hydrophobic textile materials with any selected composition, including those employed on natural fibers, cannot be predicted as further evidenced by the fact that ammonium chloride cannot be substituted for ammonium bromide in the compositions set forth herein with equivalent results.

Although it was discovered that good flame-resistance could be obtained by impregnating nylon, cellulose acetate, an acrylic fiber (copolymer of 95% acrylonitrile and 5% methyl acrylate), and the like with an aqueous solution of ammonium bromide alone, the resulting finish was nondurable, somewhat hygroscopic and tended to dust off under severe flexing or manipulation of the textile fabric. It was found that the addition of an aminotriazine condensate overcame these defects and also provided a resin finish durable to dry cleaning; accordingly, the compositions employed in the novel process comprise ammonium bromide and an aminotriazine resin. In addition to its fire-retardant qualities the salt also functions as a curing agent for the aminotriazine-aldehyde condensate; while other and more efficient accelerators may be added if desired, they are not necessary. Thus, a combined fire-retardant and resin finish is obtained on the textile which is particularly desirable for stiffening fabrics such as the nylon netting used in making underskirts and veils for women.

When the novel treatment is applied to a hydrophobic synthetic textile fabric of a flammable and fusible nature, the behavior of the treated fabric in a flame is quite different from that of the untreated fabric. The fabric will still melt while in the flame, but charred beads are formed which show little or no tendency to separate and drop off the remainder of the fabric. This substantially eliminates injury to persons from exposure to molten flaming drops of the textile material and minimizes spreading the fire. Further, the treated fabric displays no afterglow or afterflaming when the test flame is removed.

The treatment described herein is particularly applicable to the flameproofing of net materials, upholstery fabrics, draperies, stage scenery, and the like. It also provides effective results on knitted and felted fabrics as well as loose fibers which may be employed as stuffing or padding.

Aminotriazine resins suitable for use in the process described herein include water-soluble condensates of an aldehyde with an aminotriazine of the type disclosed in the Widmer et al. Patent No. 2,197,357 as well as those set forth in Johnstone et al. Patent Re. 22,566. These materials are commonly applied to cloth in textile finishing as aqueous solutions containing a curing or polymerization catalyst or accelerator; then the fabric is dried and cured at an elevated temperature to convert the resin to the water-insoluble state. Widmer et al. disclose a large number of aminotriazines including melamine, acetoguanamine, propioguanamine, melam, melem,

ammeline, ammelide, and the like, which may be condensed with various aliphatic, aromatic and heterocyclic aldehydes. Formaldehyde is of course the preferred aldehyde by reason of its cheapness and ready availability. Johnstone et al. teach that desirable textile treating resins can be manufactured from such products by alkylating them to produce the methyl or other alkyl ethers of the aminotriazine-aldehyde condensation product. Methylated trimethylol melamine and di- and trimethylol melamines are especially recommended for the present purposes. The expressions "aminotriazine resin," "aminotriazine-aldehyde condensate" and "melamine-formaldehyde condensate" are employed herein to denote both the alkylated and unalkylated derivatives disclosed in the aforesaid patents.

To provide adequate flame-resistance, the treated fabrics should contain at least about 12% of the ammonium bromide based on the dry weight of the untreated fabric and it is usually preferred to deposit 15% or more of ammonium bromide on the fabric. Where a relatively stiff fabric is desirable or acceptable, this quantity may be increased to between 20 and 50% and in some instances it may be desirable to deposit the largest quantity obtainable in an aqueous bath; thus, amounts of ammonium bromide running up to 100% or more of the weight of dry untreated fabric are suggested. The weight of resin deposited should be more than the weight of the ammonium salt; thus, for each 100 parts of ammonium bromide in the treating bath the quantity of aminotriazine resin may range from slightly more than 100 up to 500 parts or more, bearing in mind that excessive amounts of resin may limit the deposition of ammonium salts on the fabric to an extent sufficient to diminish the fire-retardant qualities of the product. In general, between about 105 and about 300 parts of resin are desirable for each 100 parts of ammonium bromide in the pad bath.

The impregnated fabrics may be cured in conventional equipment operating at temperatures ranging from about 200 to about 500° F. for periods varying inversely with the temperature and ranging from about 10 seconds to about 30 minutes. In practice, there is little need to ever exceed 400° F.

For better understanding of the nature and objects of the present invention reference should be had to the following examples in which proportions are expressed in terms of weight unless otherwise indicated.

Example 1

Nylon twill is padded through an aqueous solution of 40 parts of methylated methylolmelamine and 20 parts of ammonium bromide in solution in 40 parts of water. The squeeze rolls are adjusted for a wet pick-up of 100% based on the weight of dry cloth. The impregnated cloth is dried for 3 minutes at 220° F. and then cured for 6 minutes at 290° F. The resulting fabric does not flame when tested with a match flame.

Example 2

Example 1 is repeated with a more dilute treating bath containing 30 parts of methylated methylolmelamine and 15 parts of ammonium bromide dissolved in 55 parts of water. Here the nylon twill is bearing 30% resin and 15% ammonium bromide based on the weight of dry fabric. Again, the treated fabric does not flame when tested with a match flame.

Example 3

Nylon net is impregnated and cured in the manner set forth in Example 1. Upon applying the standard A.A.T.C.C. flame test, the char height is found to be 7.2 inches and no dripping of molten particles of nylon occurs during the test. The untreated nylon net material melts and some flaming drops fall off in this test whereas nylon net stiffened with the same quantity of the mel-

amine resin is completely combustible because of its open construction which affords ready access to air and its relatively great surface area; thus this particular material provides an extremely severe test for a flame-retardant composition.

Example 4

Nylon netting is padded through a bath made up of 30 parts of ammonium bromide, 32 parts of methylated trimethylol melamine and 38 parts of water, using two dips and two nips and a pressure of one ton; and the wet pick-up amounts to about 75%. After drying and curing for 3 minutes at 300° F. the material is subjected to a commercial dry cleaning operation and thereafter found to have a char length averaging 4.2 inches when several samples are tested by the standard test. Other samples of the treated fabric are subjected to 3 dry cleanings and then found to have an average char length of 6.5 inches in the standard flame test. Additional treated samples are subjected to 5 dry cleanings and then found to have an average char length of 4.8 inches in the flame test. None of the samples shows afterglow or afterflaming in the A.A.T.C.C. test and none supports combustion upon contact with a match flame.

Example 5

Example 4 is repeated using a bath containing 40% of the same resin, 30% ammonium bromide and 30% water with the squeeze-roll adjusted to provide 75% wet pick-up. The average char-length of the sample after one dry cleaning is 4.4 inches. Samples dry cleaned 3 times are found to have an average char length of 5.1 inches in the standard flame test and those subjected to 5 dry cleanings have an average char length measured at 5.6 inches.

Example 6

Example 4 is repeated using a pad bath containing 48% methylated trimethylol melamine, 30% ammonium bromide and 22% water with a 75% wet pick-up as before. The average char length of the impregnated fabric is 6.6 inches after 1 dry cleaning, 6.5 inches after 3 cleanings and 4.8 inches after 5 dry cleanings.

Example 7

Example 4 is again repeated with a pad bath containing 56 parts of the same resin, 30 parts of ammonium bromide and 14 parts of water with the usual 75% wet pick-up. After 1 dry cleaning of the treated fabric the average char length is found to be 3.7 inches. After 3 commercial dry cleanings, the figures is 5.6 inches, and after 5 dry cleanings the average char-length of the material is found to be 7.1 inches upon application of the standard test.

No afterglow or afterflaming is present during any of the flame tests run in Examples 5 through 7 inclusive and none of those treated samples supports combustion when tested with a burning match.

The bath used in Example 5 is stable for 10 hours without appreciable precipitation. By addition of one, two and three percent of ammonia calculated as NH_4OH , to samples of this pad bath the stability is increased to 20, 28 and 44 hours respectively. Such stability is ample for practical application of the present process in conventional textile finishing plants.

Example A

The procedure of Example 4 is followed in impregnating nylon net with an aqueous solution of 10 parts of ammonium chloride, 40 parts of methylated trimethylol melamine and 50 parts of water. No dry cleaning operations are employed. After curing the impregnated fabric is found to be combustible.

Example B

A treating bath containing 30 parts of ammonium chloride, 40 parts of the same resin and 30 parts of water is

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made up in an effort to duplicate Example 5, but the resin precipitates immediately and hence cannot be applied to a textile fabric.

Example C

Upon preparing a bath from 20 parts of ammonium chloride, 40 parts of methylated trimethylol melamine and 40 parts of water, the resin again precipitates.

Since certain changes may be made in carrying out the above process and in the product without departing from the scope of the invention, it is intended that all matter contained in the above description and examples shall be interpreted as illustrative and not in a limiting sense.

I claim:

1. A process for producing a fire-retardant nylon fabric which comprises applying an aqueous solution of 100 parts by weight of ammonium bromide and between about 100 and about 500 parts of an aminotriazine-formaldehyde condensate to said nylon fabric in sufficient quantity to deposit thereon at least about 12 percent of ammonium bromide based on the dry weight of the fabric and curing the condensate to the water-insoluble state by heating the treated fabric to an elevated temperature.

2. A process for producing a fire-retardant nylon fabric which comprises applying an aqueous solution of 100

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parts by weight of ammonium bromide and between about 100 and about 500 parts of a melamine-formaldehyde condensate to said nylon fabric in sufficient quantity to deposit thereon at least about 15 percent of ammonium bromide based on the dry weight of the fabric and curing the condensate on the fabric at a temperature between about 200 and about 400 degrees Fahrenheit for a period sufficient to convert the condensate to the water-insoluble state.

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