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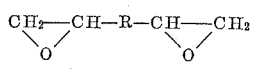
# COMPOSITION AND PROCESS FOR PRODUCING POLYESTER TEXTILE MATERIALS HAVING WATER REPELLENT AND ANTISTATIC PROPERTIES

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9 Claims. (Cl. 260—29.2)

Polyester textile fibers and fabrics are synthetic materials having widespread use in the manufacture of consumer garments and industrial fabrics. The physical properties of polyester fibers, such as "Dacron" of E. I. du Pont de Nemours and Company, "Fortrel" of the Celanese Corporation, "Kodel" of Eastman Corporation and "Vycron" of Beaunit Mills, Inc., have many advantages over those of natural or regenerated cellulosic or protein fibers and transmit these advantages to fabrics manufactured from them. However, polyester fibers and fabrics suffer from a serious disadvantage. Being hydrophobic in nature, that is, having a low tendency to absorb moisture, they have the undesirable property of accumulating electrostatic charges developed by friction during processing of the fabric and during normal industrial or consumer use of the articles manufactured from them.

Two methods have been used in the art to overcome this inherent propensity for polyester to accumulate electrostatic charges. One is the treatment of the fabrics or fibers with durable antistatic chemical agents such as are described in U.S. Patents 2,882,185, 2,914,427 and 2,982,751. These are cationic polyelectrolytes containing ethoxy and amino groups. These compounds are water-soluble polymers which are applied to polyester fabrics in aqueous solution together with a curing agent. Upon heating at temperatures above 210° F., the curing agents react with the cationic polymer to produce a cross-linked, insoluble film around the polyester fibers. The treated fabrics then possess low electrical resistance, which results in good antistatic properties, and these properties are durable to repeated laundering and dry cleaning. The curing agents which have been successfully used and described in the above-mentioned patents are polyhalides or polyepoxides. An example of a polyhalide catalyst is the polyethylene glycol diiodide (sold by Onyx Chemical Corp.) under the name "Aston Catalyst." An example of a satisfactory polyepoxide catalyst is "Eponite 100" (sold by Shell Chemical Corp.). It is a di-epoxide having the formula:



A second method used to reduce the static accumulation properties of polyester fabrics is to prepare fabrics from blends of the hydrophobic polyester fiber and hydrophyllic cellulosic fibers such as cotton and rayon. Such blended polyester-cellulosic fabrics containing up to 50% cellulosic fiber content are now sold on the market and are extensively used in the manufacture of textile products such as suits, dresses, shirts, etc.

But though it has been possible to produce polyester or blended polyester-cellulosic fabrics with acceptable antistatic properties as outlined above, it has been very difficult to produce fabrics that possess both antistatic and water-repellent properties. This is to be expected since achievement of water repellency requires the modification of the fibers or fiber surfaces to make them extremely hydrophobic and such hydrophobic fibers, having high electrical resistivity, will have poor antistatic properties. Conversely, treatment of the fibers with the



durable antistatic agents mentioned above will produce a surface containing ethoxy groups and ionic groups, both of which are hydrophyllic and tend to make such a surface easily wettable by water.

It is therefore an object of this invention to provide a novel process for finishing polyester and polyester-cellulosic blended fabrics to impart simultaneous water-repellent and antistatic properties to them.

It is a further object of this invention to provide a composition and process for finishing polyester and polyester-cellulosic blended fabrics to impart water-repellent and antistatic properties which are durable to repeated launderings and dry cleanings.

It is a further object of this invention to provide a polyester or polyester-cellulosic blended textile material which exhibits durable water-repellent and antistatic properties without affecting the physical and aesthetic properties of the fabric such as whiteness, handle and tensile strength.

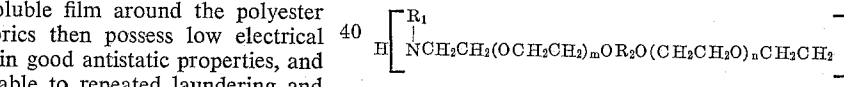
Other objects and advantages of the invention will become apparent from the following description.

The process of this invention comprises the impregnation of a polyester or polyester-cellulosic blended fabric with an aqueous composition containing the following:

(a) A cationic, polyethoxy-containing polymeric electrolyte
(b) A polyaziridinyl curing agent
(c) A water-repellent silicone resin or fluorochemical oil- and water-repellent compound,

then heating the fabric at temperatures above 220° F.

The cationic polyethoxy-containing polymeric electrolytes suitable for the practice of this invention are those mentioned above. Their structure may vary over a wide range, but they consist essentially in polymeric materials having a positive charge and containing a plurality of ethoxy and amino groups. They may be represented by the general formula:



wherein $R_1$ is the residue of a primary monoamine after the removal of the primary $NH_2$ group, $R_2$ is an organic divalent radical, $m$ and $n$ are average numbers between 3 and 40, $p$ is an integer greater than 1, and wherein X is the ester forming residue of an inorganic acid or of an organically substituted sulfuric, phosphoric, and sulfonic acid such as Cl, Br, I, $CH_3SO_3$, $OSO_3H$, and the like. The preparation of these polytertiary amines is fully described in U.S. Patent 3,070,552, issued December 25, 1962.

The water-repellent silicon resins suitable for the practice of this invention consist of mixtures of poly (dimethyl siloxanes) and poly (methyl hydrogen siloxanes) described in Moilliet, "Water-Proofing and Water Repellency," Elsevier Publishing Co., New York, pp. 153–160 (1963). Such water-repellent silicone resins, such as "Sylmer 1108" (sold by Dow Corning Corp.) are marketed as aqueous emulsions under a variety of trade names. Examples of such water-repellent emulsions are: "Repelotex HE" a 30% active water-repellent silicone resin emulsion (sold by Onyx Chemical Corp.), "Hydro Pruf" (sold by Arkansas Co.) and "Norane "Silicone" (sold by Warwick Chemical Division, Sun Chemical Co.). These emulsions of water-repellent silicone polymers are used together with an appropriate organometallic catalyst which serves to insolubilize the silicone polymer on heating. Appropriate catalysts include zinc, tin and zirconium salts of organic naphthenic and fatty acids having 8 to 18 carbon atoms and are supplied as emulsions or pastes dispersible in water and

compatible with the silicone water-repellent emulsions. Examples of such catalysts are: "Repelofix 7 S" (sold by Onyx Chemical Corp.), "Coblefix" (sold by Arkansas Co.) and "Catalyst 47" (sold by Dow Corning Corp.).

The fluorochemical water- and oil-repellent compounds suitable as a component of the composition of this invention are polymers of perfluoroalkyl acrylates and perfluoroalkyl methacrylates or copolymers of same, marketed under the trade name Scotchguard by Minnesota Mining & Mfg. Co. described in Patent 3,102,103 and also in volume 32, Textile Research Journal, pages 320–331 (1962).

The polyaziridinyl compounds necessary for the practice of this invention are organic compounds containing at least two aziridinyl groups and at least one hydrophyllic group capable of conferring water solubility or emulsifiability in water on the molecule, such hydrophyllic group or groups being non-reactive with the aziridinyl group. Examples of suitable polyaziridinyl componnds, described in my abandoned application Serial No. 268,590 filed March 28, 1963, are tris-1-aziridinyl phosphine oxide and 1,6-bis-(N-ethylenecarbamido)hexane.

The quantities of each ingredient used in the treating bath may vary over wide limits, depending upon the particular fabric to be treated and on the degree of antistatic and water-repellent properties required by the end-use of the treated fabric. Preferred ranges of composition for the practice of this invention are as follows, the amounts being expressed as percentages by weight of active material present in the aqueous treating bath:

(a) 1.0 to 5.0% of antistatic agent
(b) 0.2 to 2.0% of polyaziridinyl curing agent
(c) 1.0 to 3.0% of silicone polymer

together with 0.3 to 1.5% of silicone curing catalyst, or 1.0 to 3.0% of fluorochemical compound.

The fabric to be treated is passed through the treating bath containing the above ingredients in a padder or similar textile-impregnation equipment, then heated at elevated temperatures on drying cans or in a curing oven. The time and temperature of heating may be varied over wide limits, a preferred range being 1 to 5 minutes at about 250° F. to about 350° F.

This invention will be further described by the following examples which are presented as illustrative and are not to be considered as limiting the practice of the invention. In the examples described below, launderings were carried out in a Westinghouse commercial Laundromat using Tide (sold by Procter and Gamble Co.) detergent and the "hot" (140° F.) setting with a five-pound load, followed by ironing on a flatbed press. Dry cleaning was carried out by tumbling the fabric in Stoddard solvent at room temperature for 1 hour, followed by air drying. Water repellency was evaluated by AATCC Spray Test Method 22–1961, Technical Manual of the American Association of Textile Chemists and Colorists, volume XXXVIII, page B 119. Antistatic properties were evaluated by the AATCC Electrical Resistivity Method 84–1960, page B 103 in the above-mentioned manual. Water repellency was considered satisfactory if the spray rating was a minimum of 90 initially and 70 after 5 launderings or dry cleanings. Antistatic properties were considered satisfactory if the electrical resistivity after 5 launderings or dry cleanings, expressed as Log R, at 30% relative humidity and 75° F., was less than 13.0. Oil repellency was evaluated by the method described in Scotchguard Technical Bulletin, Appendix A., Minnesota Mining & Mfg. Co. (1959), and considered satisfactory if ratings were 90 minimum.

*Example 1*

An aqueous bath was prepared containing 27% Aston 123 (sold by Onyx Chemical Corp.) (20% active), 2.8% Chemirad APO (sold by Chemirad Corp.) (an 85% alcoholic solution of tris-1-aziridinyl phosphine oxide), 6% Repelotex HE (sold by Onyx Chemical Corp.) (a 30% active water-repellent silicone emulsion) and 2% Repelofix 7 S (sold by Onyx Chemical Corp.) as curing agent for the silicone emulsion. A white poplin fabric consisting of 65% Dacron polyester fiber and 35% cotton (Testfabrics, Inc., Style #7402) was passed through this aqueous solution in a 3-roll padder, using a roll pressure which allowed the fabric to pick up approximately 50% of its own weight of treating solution. The fabric was then framed and heated 2 minutes at 220° F., then 5 minutes at 300° F. in an electric circulating-air oven. The sample was designated as Sample A and its properties are tabulated in Table I.

*Example 2*

The process of Example 1 was repeated, except that the curing agent Chemirad APO was replaced by 2% Eponite 100 (sold by Shell Chemical Corp.) mixed with 0.5% Neutronyx 600 (sold by Onyx Chemical Corp.), a condensation product of nonyl phenol with 9.5 moles of ethylene oxide. This sample was designated Sample B.

*Example 3*

An aqueous treating bath was prepared containing 10% Aston 108 (sold by Onyx Chemical Corp.), 1% Chemirad APO and 8.6% Scotchguard FC 208 (28% active) (sold by Minnesota Mining & Mfg. Co.). A small amount of hydrochloric acid was added to reduce the pH value of the solution to 5.0. White Dacron taffeta (Testfabrics, Inc., Style #709) was treated with this solution in a 3-roll padder with the roll pressure adjusted to allow the goods to pick up approximately 25% of its weight of treating solution. The goods were then framed and heated for 2 minutes at 220° F. and 5 minutes at 300° F. The sample was designated as Sample C.

*Example 4*

The process described in Example 3 was repeated, except that the Chemirad APO was replaced by 1% Aston Catalyst (sold by Onyx Chemical Corp.) and the adjustment of the pH was eliminated. The sample was designated Sample D.

TABLE I

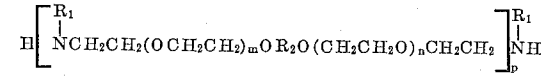
| Sample | A | B | C | D |
|---|---|---|---|---|
| Spray Rating | 90 | 0 | 90 | 90. |
| Spray Rating after 5 Launderings. | 70 | 0 | 80 | 70. |
| Spray Rating after 5 Dry Cleanings. | | | 70 | 70. |
| Log R | 10.1 | | 9.9 | 9.7. |
| Log R after 5 Launderings. | 11.7 | | 12.3 | 10.3. |
| Log R after 5 Dry Cleanings | | | 10.3 | 10.6. |
| Oil Repellency | | | 100 | 100. |
| Oil Repellency after 5 Launderings. | | | 90 | 90. |
| Oil Repellency after 5 Dry Cleanings. | | | 90 | 90. |
| Handle | Soft | Slightly Firm. | Soft | Slightly Firm |
| Yellowing | None | None | None | Marked. |

The data tabulated above clearly demonstrate that the novel compositions of this invention and the finishing process described impart simultaneous durable water-repellent and antistatic properties to polyester and blended polyester-cellulosic fabrics. Furthermore, the data show that the novel use of polyaziridinyl curing agents in the compositions of this invention permit the achievement of the desirable water-repellent and antistatic properties without impairing the aesthetically important properties of soft handle and whiteness, an achievement which has not been possible with processes heretofore known to the art which utilize polyhalide or polyepoxide curing agents.

I claim:

1. A process for simultaneously imparting antistatic and water-repellent properties to polyester and blended polyester-cellulosic blended fabrics which comprises the steps of impregnating the fabric with an aqueous anti-

static composition containing an antistatic agent comprising a water-soluble cationic polyelectrolyte containing ethoxy and amino groups, having the general formula



wherein $R_1$ is the residue of a primary monoamine after removal of the primary $NH_2$ group, $R_2$ is an organic divalent radical, *m* and *n* are average numbers between 3 and 40, and *p* is an integer greater than 1, a polyaziridinyl curing agent for said antistatic agent, and a water-repellent agent selected from the group consisting of (1) an aqueous emulsion of a water-repellent silicone resin together with an organo-metallic catalyst for said silicone resin and (2) a water- and oil-repellent compound selected from the group consisting of polymers of perfluoroalkyl acrylates, perfluoroalkyl methacrylates and copolymers of same, and then drying the treated fabric at temperatures from about 250° F. to about 350° F. for about 1 to about 5 minutes.

2. The process of claim 1 in which the antistatic curing agent is tris-(1-aziridinyl) phosphine oxide.

3. The process of claim 1 in which the curing agent is 1,6-bis-(N-ethylenecarbamido) hexane.

4. The process of claim 2 in which the water-repellent agent is a water-repellent silicone resin.

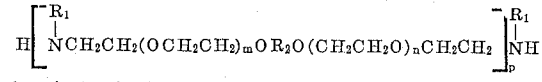
5. The process of claim 2 in which the water-repellent agent is a water-and-oil repellent compound selected from the group consisting of polymers of perfluoroalkyl acrylates, perfluoroalkyl methacrylates and copolymers of same.

6. The process of claim 1 in which the impregnating bath is an aqueous composition containing 1.0% to 5.0% antistatic agent, 0.2% to 2.0% of a polyaziridinyl curing agent, 1.0% to 3.0% of water-repellent silicone resin, and 0.3% to 1.5% of an organo-metallic silicone resin catalyst, said percentages being in terms of percentages of active ingredient in the treating bath.



7. The process of claim 1 in which the impregnating bath is an aqueous composition containing 1.0% to 5.0% antistatic agent, 0.2% to 2.0% curing agent, and 1.0% to 3.0% water-and-oil repellent compound selected from the group consisting of polymers of perfluoroalkyl acrylates, perfluoroalkyl methacrylates and copolymers of same.

8. A composition for imparting water-repellent and antistatic properties to polyester and blended polyester-cellulosic blended materials consisting of an aqueous mixture of a water-soluble polyelectrolyte containing ethoxy and amino groups having the general formula



wherein $R_1$ is the residue of a primary monoamine after removal of the primary $NH_2$ group, $R_2$ is an organic divalent radical, *m* and *n* are average numbers between 3 and 40, and *p* is an integer greater than 1, a polyaziridinyl curing agent, and a water-repellent agent selected from the group consisting of (1) water-repellent silicone resins together with organo-metallic catalysts for said silicone resin and (2) a water-and-oil repellent compound selected from the group consisting of polymers of perfluoroalkyl acrylates, perfluoroalkyl methacrylates and copolymers of same.

9. Polyester and blended polyester-cellulosic fabrics treated by the process of claim 1.


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