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(54) Antisoil nylon fibers.

(57) Antisoil nylon fibers are provided by first coating the fibers with a polyacrylic acid and then with a fluorochemical. The polyacrylic acid chemically bonds to the surface of the fibers and to the fluorochemical. The resulting coating has greater adherence to the fiber than does corresponding coatings from which the polyacrylic acid is omitted.

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ANTISOIL NYLON FIBERS

BACKGROUND OF THE INVENTION

A. Field of the Invention

This invention relates to processes for imparting water and oil repellency properties to nylon fibers by applying fluorochemicals particles to the surface of the fibers from aqueous dispersions thereof. More particularly, the invention relates to an improvement in such processes whereby the fluorochemical particles are more tenaciously attached to the nylon surface.

B. Description of the Prior Art

Fluorochemicals, such as, Scotchgard fluorochemicals (a brand name of Minnesota Minning and Manufacturing Company) and Zepel and Teflon fluorochemicals (brand names of duPont), are industrially used to provide fiber surfaces with water and oil repellency properties, i.e., antisoiling properties. In the prior art processes, fluorochemicals are used in the form of aqueous dispersions having a particle size ranging from 200 to 1000 angstroms in diameter. The particles are conventionally dispersed by means of an emulsifier which may impart a charge to the particles. Fibers are treated with the dispersions and then dried leaving a coating of the fluorochemical particles on the fiber surface. The bonding (or fixation) of the particles to the surface is mainly by the van der Waals and Coulombic forces between the fluorochemical particles and the fiber surface, thereby limiting the strength of the bonding to 5 to 10 Kcal./mol bonding. Consequently, fluorochemical losses from the coated fibers during the dyeing of the fibers or fabrics made therefrom (e.g. carpeting) are often substantial.

SUMMARY OF THE INVENTION

The present invention relates to an improvement in the above-mentioned prior art processes for using fluorochemicals to impart antisoiling properties to nylon fiber surfaces. The improvement enhances the fixation of the coating of fluorochemical particles to the fiber surfaces and comprises the preliminary step of coating the fibers with a material which chemically bonds to the fiber surfaces and which will bond to positively charged fluorochemical particles when brought into contact therewith. The material functions as an interlayer between the surface of the fibers and the coating of fluorochemical particles. Nylon fibers coated with fluorochemical particles in accordance with the improvement of the present invention have more durable and longer lasting antisoiling properties; the fixation of the fluorochemical particles to the fiber surface is enhanced as compared to corresponding coatings in which the interlayer is omitted.

PREFERRED EMBODIMENTS OF THE INVENTION

In practicing the improvement of the present invention nylon fibers are first coated with an interlayer material and then with a fluorochemical. The interlayer material forms a chemical bond with the nylon surface of the fibers and with the fluorochemical.

Any nylon fiber may be treated in accordance with the improvement of the present invention. The term "nylon fiber" as used herein means a fiber in which the fiber-forming substance is any long chain synthetic polyamide having recurring amide groups (-NH-CO-) as an integral part of the polymer chain. Such substances include nylon 66, which is polyhexamethylene adipamide, and nylon 6, which is polycaprolactam. The fiber may be in the form of a continuous filaments or a yarn composed of continuous filaments or a yarn composed of staple.

Materials which may be used to form the interlayer coating in accordance with the improvement of the invention are materials which are capable of chemically bonding to positively charged fluorochemical particles and to the surface of nylon fibers. Representative such materials include, polyacrylic acid (PAA) and polymethacrylic acid (PMA) and copolymers of acrylic or methacrylic acid with one or more vinyl monomers copolymerizable therewith such as vinyl acetate, methylacrylate, methacrylate, styrene, vinyl chloride, vinylidene chloride and the like. The copolymers are composed of at least 10% and, preferably, at least 25% by weight of the acrylic or methacrylic acid. The interlayer material is preferably applied to the nylon fibers from an aqueous medium, and then the fibers dried before they are treated with the fluorochemicals; otherwise, the fluorochemicals tend to attach to the nylon fiber surface before the interlayer material has had a chance to react with the nylon fiber surface. The fibers may be dried under ambient conditions or may be heated to shorten the drying time. Preferably, the material is applied to the fibres from an aqueous medium comprising from $5X10^{-3}$ to $5X10^{-2}$ moles per litre of the material.

Fluorochemicals which may be used in practicing the improvement of the invention include those containing one or more fluorinated aliphatic radicals having from 3 to 20 carbon atoms and having a carbon-bonded fluorine content of 40-78% by weight. Preferably, the radical is perfluoro alkyl radicals of the general formula $-C_nF_{2n+1}$. Generally, the fluorochemicals will contain 10 to 60% by weight of carbon-bonded fluorine. The fluorochemical are preferably applied to the nylon fibers from aqueous dispersions of positively charged fluorochemical particles. Such aqueous dispersions are prepared by dispersing the fluorochemical particles in an aqueous medium using emulsifiers such as quaternary ammonium salts as the dispersing agent. The emulsifier imparts a positive charge to the particles. Fluorochemicals, such as, Scotchguard, Zepel and Teflon fluorochemicals are already commercially available in the form of an aqueous dispersion of positively charged fluorchemical particles. The dispersion preferably comprises from 0.5 to 2.0% by weight of fluorine.

The following examples are given to further illustrate the invention. In the examples, parts and percentages are by weight.

EXAMPLE 1

Skeins of continuous filament nylon 66 carpet yarn having a total denier of about 1300 and being composed of 68 filaments were impregnated with an aqueous solution of PAA having an average molecular weight of 250,000. After centrifugation and air drying for 2 hours, the skeins were immersed in aqueous dispersions of commercially obtained fluorochemicals, i.e., either Scotchgard fluorochemical designated FC-391 or L-5111. After contrifugation, each skein was divided into two yarns of approximately equal length; one yarn was vacuum dried over night and analysed for fluorine; the other yarn was blank-dyed in an aqueous medium for 75 minutes at the boil (100°C.), vacuum dried over night and then analysed for fluorine. The concentration of fluorine [F] on the fiber surface before blank-dyeing, expressed in parts per million (ppm), and the amount of fluorine retained after blank-dyeing, expressed as a percentage are given in the table below. The concentration of PAA [PAA] in the aqueous solution and the concentration of fluorine [F] in the aqueous dispersion of the fluorochemical is also given in the table.

TABLE

Item	[PAA] (moles/ liter)	Fluoro-Chemical	[F] %	[F] on Fiber surface (ppm)	F-retention %
1	none	L-5111	1.0	240	82
2	10^{-3}	L-5111	1.0	1000	79
3	10^{-2}	L-5111	1.0	1509	100
4	10^{-3}	L-5111	1.0	8800	78
5	none	FC-391	0.67	150	60
6	10^{-3}	FC-391	0.67	206	65
7	10^{-2}	FC-391	0.67	565	83
8	10^{-1}	FC-391	0.67	1060	52

The data in the table show the influence of PAA on fluorine uptake, as well as on F-retention after blank-dyeing. For the cationic fluorochemicals used, the uptake increased with [PAA], whereas the retention had a maximum at a [PAA] of 10^{-2} moles/liter. If the ratio of [PAA]/[F] is too low there are not enough COOH groups available to bond with the positive charges of the fluorine particles, and the F-retention is low. If the ratio is too high, part of the PAA molecules are not bonded to the nylon surface of the fiber and are washed off during dyeing, taking with them fluorine particles which are bonded thereto. For the fluorine concentration [F] used in this example (% F in the aqueous fluorocarbon dispersion), a [PAA] of about 10^{-2} moles/liter appears to give best fluorine retention. When using different fluorine concentrations, experiments such as those shown in the above table can be carried out to determine optimum polyacrylic acid concentrations.

In related experiments similar results were obtained when using polymethacrylic acid instead of polyacrylic acid.

In accordance with a preferred embodiment of the invention, the nylon continuous filament yarn is treated during its melt spinning operation by passing the yarn after it is quenched over two inline applicator rolls before the yarn is collected. The first roll applies the interlayer (e.g. PAA) to the surface of the yarn fibers and the second roll applies the aqueous dispersion of positively charged particles of fluorochemical to the interlayer. The yarn is dried after application of the interlayer material and before application of the fluorochemical by passing it over a heated roll. The yarn may be dried by passing it around a heated roll, for example, around a heated separator roll and its associated idler roll with one or more wraps.

What we claim is :

1. In a process for coating the surface of nylon fibers with fluorochemical particles to enhance the water and oil repellency of the fibers wherein the fibers are treated with an aqueous dispersion of positively charged colloidal particles of the fluorochemical(s) and then dried to leave the fluorochemical particles on the surface of the fibers, the improvement of enhancing the fixation of the particles to the surface of the fibres which comprises the preliminary step of coating the fibers with a material which chemically bonds to the surface of the fibers and will chemically bond to the fluorochemical particles when brought into contact therewith.

2. A process of Claim 1, wherein the material is applied to the fibers from an aqueous medium, and the fibers are dried before they are treated with the fluorochemical.

3. A process of Claim 1 or Claim 2, wherein the material comprises polyacrylic acid.

4. The process of Claim 1 or Claim 2, wherein the material comprises polymethacrylic acid.

5. A process according to any of the preceding claims, wherein the material is applied to the fibers from an aqueous medium comprising from 5×10^{-3} to 5×10^{-2} moles per litre of the material.

6. A coated nylon fiber characterized in that the coating comprises a material chemically bonded to the surface of said fiber and fluorochemical particles chemically bonded to the material.

7. A coated fiber of Claim 6, wherein the material comprises polyacrylic acid.

8. A coated fiber of Claim 6, wherein the material comprises polymethacrylic acid.