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3,541,075

METHOD OF PRODUCING SOIL RESISTANT FIBERS

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4 Claims

ABSTRACT OF THE DISCLOSURE

Acrylic fibers are rendered soil resistant in a continuous wet spinning process wherein the coagulated fibers are washed and stretched, passed through a first finish bath containing standard textile processing finishes, partially dried, impregnated with a second finish composition having anti-soil properties, and essentially completely dried to produce a textile fiber having durable soil resistant properties.

BACKGROUND OF THE INVENTION

Field of the invention

This invention relates to the manufacture of synthetic textile fibers, and specifically deals with the continuous manufacture of acrylic fibers which are resistant to soiling by virtue of a soil resistant finish applied thereto.

Description of the prior art

In the textile field, and particularly in the carpet industry where the popularity of light color shades and the use of carpets in entrance ways have increased in recent years, the need for soil resistant fibers has become more evident. The textile industry has responded to this need with extensive research into the area of special textile finishes which impart the desired soil resistant properties to the synthetic fibers. One of the most effective finishes developed incorporates a zirconium salt in an acidic aqueous solution, preferably containing as an additional ingredient a nitrogen containing base. Such textile finishing compositions are described in detail in the copending applications having Ser. Nos. 564,003 and 564,041, both filed July 11, 1966. It was discovered that the treatment of textile articles such as woven cloth or tufted carpets with these specific finish compositions imparted a high degree of soil resistance to the article.

For reasons of convenience and economics, it is desirable to apply the zirconium containing finish composition to the synthetic fibers during the manufacture thereof in preference to treating the finished textile goods. In the production of acrylic fibers by conventional wet spinning techniques, a standard fiber finish comprised of various lubricating, softening and/or antistatic agents is applied to the spun fiber from an aqueous bath after the fiber has been washed free of residual solvent. After the application of finish, the fiber is dried by passing it around and over heated drying rolls. However, when the soil resistant finish composition is incorporated into the standard finish bath, production is disrupted by broken filaments and solids deposits on the drying rolls. These difficulties make the commercial production of a soil resistant fiber by this technique impossible.

SUMMARY OF THE INVENTION

This invention overcomes the difficulties pointed out above by applying a soil resistant finish to acrylic fibers

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produced by conventional wet spinning techniques through first applying to the washed fibers a conventional textile finish, partially drying the fibers, then applying the soil resistant finish, and finally drying the fibers to essentially complete dryness.

The conventional finish may be applied to the fiber by passing the fiber through an aqueous bath. The fiber, then containing about 70% moisture at ambient temperature, is heated sufficiently to increase the temperature to above about 70° C. and to reduce the moisture content to between about 55 and 5% and the soil resistant finish is then applied to the hot fiber. The fiber is finally dried to less than about 0.5% moisture content and processed in accordance with conventional techniques to produce tow or staple for conversion into textile articles.

The fibers treated with the dual finish system may be dried by passing over and around hot drying rolls with no appreciable solids deposition or filament breakage occurring. The resulting fibers possesses significantly greater resistance to soilage than fibers treated with the conventional textile finish alone. The method of this invention provides for the continuous production of soil resistant fibers in a commercial manner with only minor changes required of the conventional spinning equipment.

It is therefore an object of this invention to provide a method for the continuous production of soil resistant synthetic fibers.

Another object of this invention is to provide a method for the continuous production of acrylic fibers which possess a durable soil resistant finish without associated broken filaments and solids deposits.

These and other objects and advantages will become more readily apparent when read in conjunction with the remainder of the specification and considered along with the included examples.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

According to a preferred embodiment of the present invention, an acrylic fiber is wet spun according to conventional techniques wherein a spinning solution comprised of polymer and solvent is extruded through a spinning orifice into a coagulation bath comprised of water and solvent. The coagulated filaments are stretched to achieve molecular orientation, and washed free of residual solvent.

The wet spinning technique is a well known process for the production of synthetic fibers. The acrylic fibers which may be produced in this manner include, in addition to polyacrylonitrile, a wide range of copolymers and terpolymers which contain a preponderance of acrylonitrile. Such polymeric compositions are described in detail in U.S. Pat. 3,318,983.

After washing, the spun tow is passed through a bath where conventional textile finishing compounds are applied to the fibers. These compounds are necessary to assure that the fiber will process well on textile machinery when being converted into yarns and fabrics. One example of such a conventional textile finishing bath is an aqueous solution containing 2 percent of a textile lubricating agent comprised of 60 percent sorbitan nonopalmitate and 40 percent castor fatty acid with 200 moles ethylene oxide, and 1 percent of an antistatic agent such as soya dimethylaminoethyl ethosulfate. The finish may in addition contain textile softening agents if desired, and other lubricating and antistatic agents may be substituted

for those specifically mentioned above. Such agents are well known and widely used throughout the textile industry.

As the tow leaves the finish bath, it is passed between two wiper bars to remove excess finish solution and thence onto the drying rolls. At this point, the fibers in the tow normally contain between 60 and 70 percent moisture and are at ambient temperature of about 25 to 30° C.

On the drying rolls, the tow is laced between an upper horizontal row of rolls and a lower horizontal row of rolls, passing alternately over an upper roll and under a lower roll in a serpentine path. Each roll is electrically or steam heated to a temperature of at least about 130° C. and preferably about 150° C., although higher or lower temperatures may be used. As the tow passes around each heated roll, part of the moisture in the tow is driven off, the exact amount being a function of roll temperature and tow contact time and area.

In a preferred embodiment of the present invention, the tow is passed around 2 to 8 pairs of heated rolls, and preferably around 4 to 6 pairs of rolls heated to between 155° C. and 165° C. and operating at a speed such that the moisture in the tow is reduced to between 55 and 5 percent while the surface temperature of the tow is heated to at least about 70° C. and preferably to above 80° C. The heated fibers are then impregnated with the soil resistant finish of the composition as hereinbefore described. This finish is conveniently applied to the tow as a spray, although a pad bath may be used with good results.

It is critical to the successful practice of this invention that the process steps as described above be carried out in the order described. Specifically, it is necessary that the conventional textile finish be applied first to the fiber, that the fiber then be partially dried, and that the soil resistant finish be applied to the hot fiber. After the application of the soil resistant finish, the tow is laced over an additional series of heated rolls to finally dry the fiber and reduce the fiber moisture content to less than about 0.5 percent. The final drying operation also causes any voids within the fiber commonly associated with wet spinning to collapse and thereby increase fiber density.

The dried fiber is then processed on into yarns and textile goods using established procedures. For example, the dried fiber may be crimped, steam relaxed, cut to staple, and converted into yarn using standard cotton or wool processing machinery. If the fiber is of a high denier, such as about 15 denier, it can be processed on woolen machinery to form yarns suitable for tufting or weaving into high quality carpets. Carpets made from such yarns spun in accordance with this invention possess markedly superior resistance to soiling, and are also easier to clean when soiled.

The effectiveness of the method of this invention in imparting soil resistant properties to synthetic fibers is illustrated by preparing tufted carpet samples from the treated fibers and subjecting these samples to a standard laboratory soiling procedure. Specifically, samples of carpet 2" by 4" are measured for initial reflectance, soiled with an artificial soil and cleaned by vacuuming, and finally measured for soiled reflectance.

The difference in the reflectance readings indicate the anti-soiling properties or characteristics of the samples, i.e. a large number indicates poor anti-soiling properties whereas a small number indicates good anti-soiling properties. The photo reflectance readings are measured by using a Photovolt Reflectance Meter (Model No. 610, Photovolt Corp., New York, N.Y.). The samples of the textile articles are soiled by either of two methods, i.e. by an artificial soiling test or by a floor soiling test. The artificial soiling test is accomplished by placing the samples in a one gallon wide mouth jar having therein two glass rods (5¾" long by ⅜" diameter) glued on opposite sides of the jar and containing 10% (based on weight of the sample) of artificial soil and 10 number

two rubber stoppers. The artificial soil has the following composition (the soil is sifted through a 30 mesh screen):

	Percent
Peat -----	22.3
5 Silica gel -----	2.1
Cement -----	2.1
Kaolin clay (peerless) -----	2.1
Molacco furnace black -----	0.2
Red iron oxide -----	0.1
10 Sawdust -----	52.5
Calcium carbonate -----	10.9
Animal charcoal -----	6.6
Mineral oil -----	1.1
15 Total -----	100.0

The jar is then sealed, placed on a ball mill and rotated at 95-100 r.p.m. for 30 minutes in a clockwise direction and 30 minutes in a counter-clockwise direction at the same r.p.m. Thereafter the samples are removed from the jar, vacuumed, and the photo reflectance readings of the two samples measured. The difference between the average initial reflectance readings and the average of the final reflectance readings indicate the degree of soil resistance. As mentioned earlier, a small photo reflectance difference number indicates a good anti-soiling property whereas a large number indicates a poor anti-soiling property of the textile article.

Control samples, that is samples which have been treated only with the conventional finish, are soiled under the same conditions as the test samples.

To confirm the results of the laboratory soiling test, a floor soil test was also conducted for selected samples. The procedure of evaluation was the same as for the artificially soiled samples except that the samples are soiled by placing them in the pathway of a well travelled walk way in order that they might be soiled by natural means. The validity of the laboratory soiling technique was confirmed by the actual floor tests.

In order that the present invention may be fully understood, the following examples are given primarily by way of illustration.

EXAMPLE I

45 Acrylic fibers of 15 denier were wet spun by extruding a spinning solution comprised of 26 parts of a polymer consisting of 93 percent acrylonitrile and 7 percent vinyl acetate, 74 parts of dimethylacetamide solvent, and 0.45% based on polymer weight of TiO₂ delusterant through a spinnerette into an aqueous coagulation bath containing 50 55 percent solvent at 40° C. The filaments were passed from the coagulation bath into a boiling water cascade where they were stretched 6 times and simultaneously washed essentially free of solvent. The filaments were then passed through a bath containing an aqueous solution of a conventional finish comprised of 66 percent of a lubricating agent containing 60% sorbitan monopalmitate and 40% castor fatty acid with 200 moles of ethylene oxide, and 34 percent of an antistatic agent consisting of 60 65 soya dimethylaminoethyl etho sulfate. After leaving the finish bath and being stripped of excess finish, the filaments were passed around 4 pairs of drying rolls heated to about 158° C. with 70 lb. steam. The moisture content of the fibers was reduced from about 70 percent when leaving the finish bath to about 20 percent when leaving the fourth roll, and surface temperature of the fiber was increased to 83° C.

As the tow proceeded from the fourth roll, it was sprayed with an excess of a second finish containing in aqueous solution a combination of the conventional finish composition described above and a soil resistant finish comprised of zirconium acetate and sulfamic acid. The tow then proceeded over the remainder of the drying rolls where its moisture content was reduced to about 75 0.2 percent.

The amount of finish applied to the fiber from the conventional bath and from the spray was varied to determine the effectiveness of the treatment at various levels. The levels evaluated are shown for each condition in Table I.

After being crimped and steam relaxed, the two was cut to 4 inch staple and processed into carpet yarn on woolen textile machinery. The yarn were finally tufted into carpet samples which were submitted to the laboratory soiling test previously described in order to determine the effectiveness of the finish treatment. The results of this evaluation are given in Table I below.

EXAMPLE IV

The process of Example I was repeated except that the spinning solution was comprised of 16.2 parts of a polymer consisting of 88.7 percent acrylonitrile, 5.3 percent vinylacetate, and 6.0 percent methyl vinyl pyridine, 2.4 parts of polyvinylchloride, and 81.4 parts dimethylacetamide solvent. The conventional finish (C.F.) was an aqueous solution of the lubricating agent only, the anti-static agent being emitted for this example. Soil resistant carpet samples were produced and evaluated for a wide range of finish application levels with the results appearing in Table IV.

TABLE IV

Sample	Percent finish applied—o.w.f.				Carpet reflectance reading			
	C.F. bath	From spray			Before soiling	After soiling	Percent soiled	Percent improvement
		C.F.	ZrAc	S.A.				
Control.....	0.7	-----	-----	-----	64	27	58	-----
1.....	0.1	0.25	0.05	1.25	61	30	51	12.1
2.....	0.5	0.25	0.05	1.25	65	32	51	12.1
3.....	0.1	1.0	0.05	1.25	68	34	50	13.8
4.....	0.5	1.0	0.05	1.25	66	37	44	23.0
5.....	0.1	0.25	0.2	1.25	68	33	52	10.4
6.....	0.5	0.25	0.2	1.25	67	34	49	15.9
7.....	0.1	1.0	0.2	1.25	67	34	49	15.9
8.....	0.5	1.0	0.2	1.25	67	35	48	17.2

TABLE I

Sample	Percent finish applied—o.w.f.					Carpet reflectance reading			
	C.F. bath	From spray			Spray pH	Before soiling	After soiling	Percent soiled	Percent improvement
		C.F.	ZrAc	S.A.					
Control.....	1.0	-----	-----	-----	-----	70	39	45	-----
1.....	1.0	1.0	0.2	1.25	^a 1.5	76	44	41	7.2
2.....	1.0	1.0	0.2	1.25	^b 1.5	72	43	41	8.0
3.....	1.0	1.0	0.2	1.25	^a 2.0	76	44	43	4.0
4.....	0.25	1.0	0.4	1.25	1.1	72	42	42	4.9

^a pH adjusted with trisodium phosphate.

^b pH adjusted with sodium metasilicate.

NOTE: C.F.=Conventional finish; ZrAc=Zirconium acetate; S.A.=Sulfamic acid; o.w.f.=on weight of fiber.

EXAMPLE II

The process of Example I was repeated under substantially the same conditions described in Example I except that a different polymer batch resulted in a uniformly lower level of carpet reflectance readings as shown in Table II.

TABLE II

Sample	Percent finish applied—o.w.f.					Carpet reflectance reading			
	C.F. bath	From spray			Spray pH	Before soiling	After soiling	Percent soiled	Percent improvement
		C.F.	ZrAc	S.A.					
Control.....	1.0	-----	-----	-----	-----	65	31	52	-----
1.....	0.5	0.5	0.1	1.25	0.7	68	34	50	3.8
2.....	1.0	1.0	0.2	1.25	0.7	70	39	44	14.8
3.....	1.0	1.0	0.2	^a 1.25	4.2	72	37	49	6.5

^a Ammonium sulfamate substituted for the sulfamic acid.

EXAMPLE III

The process of Example I was repeated except that the spinning solution was comprised of 25 parts of an acrylic polymer containing 89.8% acrylonitrile, 7.5% vinyl acetate, and 2.7% vinyl bromide, and 75 parts of dimethylacetamide solvent. The soil resistant finish was applied to the fiber at two different concentrations as shown in Table III. Carpet reflectance readings were taken before and after laboratory soiling to measure propensity of the various yarn samples according to the method of Example I, and the results of the test are shown in Table III below.

TABLE III

Sample	Percent finish applied—o.w.f.					Carpet reflectance reading			
	C.F. bath	From spray			Spray pH	Before soiling	After soiling	Percent soiled	Percent improvement
		C.F.	ZrAc	S.A.					
Control.....	0.8	-----	-----	-----	-----	62	29	53	-----
1.....	0.8	1.0	0.2	1.25	^a 1.5	73	38	49	8.8
2.....	0.8	1.0	0.4	1.25	1.2	70	37	47	11.3

^a pH adjusted with sodium metasilicate.

The above data show the excellent results obtained by applying as little 0.05% zirconium acetate to the fiber by the method of this invention.

Although the description of the instant invention has been directed primarily toward the treatment of acrylic fibers which are generally defined as those fibers wherein

acrylonitrile constitutes at least 85 percent of the fiber forming constituents, the invention is not limited thereto, but is also applicable to the treatment of wet spun modacrylic fibers. The modacrylic fibers generally contain at least 60 percent acrylonitrile and up to 40 percent of one or more other copolymerizable monomers designed to impart special properties to the fiber. For example, it is known to copolymerize acrylonitrile with vinylidene chloride, vinyl bromide, or other halogen containing monomers in order to produce a fiber having a high degree of flame resistance.

It is also contemplated that other compounds may be substituted for the ingredients of the soil resistant finish as described, and such substitution is considered to be within the scope of the invention describing a novel method for treating tows.

The foregoing illustrates the essential features of the invention as well as some of the manners in which it may be practiced. Various changes and modifications may be made in practicing the invention without departing from the spirit and scope thereof.

What is claimed is:

1. A method of imparting soil resistance to textile fibers made from an acrylonitrile copolymer comprising a major proportion of acrylonitrile and a copolymerizable portion of vinyl acetate after said fibers have been extruded, coagulated, washed, stretched and have had a textile finish applied thereto, comprising the steps of:

- (a) heating the fiber to a surface temperature of at least about 70° C. and reducing the moisture content thereof to between about 5 and 55 percent;
- (b) applying to the heated fibers a finish composition containing zirconium acetate and sulfamic acid; and
- (c) drying the fiber to a moisture content of less than about 0.5 percent.

2. A method according to claim 1 wherein the finish composition is sprayed onto the fibers.

3. A method according to claim 1 wherein the fibers contain at least 80 percent acrylonitrile.

4. A method according to claim 1 wherein the fibers are heated to a temperature above 80° C.

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