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Jenkins et al.

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[45] **Apr. 15, 1975**

[54] **ANTISTATIC POLYCARBONAMIDE COMPOSITION**

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Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 400,756, Sept. 26, 1973, abandoned.

[52] **U.S. Cl.** ..... **260/78 S;** 57/140 R; 260/28; 260/857 PG

[51] **Int. Cl.** **C08g 20/38**

[58] **Field of Search** ..... 260/78 S, 78 R, 857 PG, 260/18 N

[56] **References Cited**

UNITED STATES PATENTS

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Attorney, Agent, or Firm—John W. Whisler

[57] **ABSTRACT**

Polycarbonamide suitable for melt spinning into multifilament fusion-free antistatic yarn having dispersed therein a polyalkoxylated triglyceride of a saturated fatty acid and an alkylene bis-alkyl carboxamide. The fusion-free multifilament yarn therefrom has enhanced properties and is particularly useful as a textile yarn.

5 Claims, No Drawings

ANTISTATIC POLYCARBONAMIDE COMPOSITION

The application is a continuation-in-part of our application Serial No. 400,756, filed Sept. 26, 1973, now abandoned.

BACKGROUND OF THE INVENTION

The term nylon is used herein to describe any fiber-forming polycarbonamide which has recurring amide groups as an integral part of the main polymer chain, and wherein such groups are separated by at least two carbon atoms.

Under conditions of normal use annoying and sometimes hazardous electrostatic charges tend to accumulate on the surface of nylon fabrics. It is known from U.S. Pat. No. 3,388,104 that these electrostatic charges can be significantly reduced by incorporating into the nylon prior to filament formation from 1 to 20 percent, based on the weight of nylon, of a polyalkoxylated triglyceride of a saturate fatty acid. Typically, nylon **66** (polyhexamethylene adipamide) textile yarn is produced using a conventional spinning machine in which molten nylon **66** is extruded through a multi-orifice spinneret to yield fusion-free filaments that are cooled, converged and subsequently packaged as a multifilament yarn in a conventional manner. However, when molten nylon **66** containing said triglyceride dispersed therein is extruded into filaments using this same conventional spinning machine, two or more of the filaments of the resulting yarn are randomly fused. Random fusion of yarn filaments causes a significant reduction in the quality and appearance of the yarn when the yarn is subjected to subsequent processing operations. For example, fusion of yarn filaments causes a significant reduction in the drawtwist performance of the yarn. Drawtwist performance is a measure of the quality of drawn yarn and is generally expressed in terms of the number of yarn breaks per pound of drawn yarn.

In addition to poor drawtwist performance, fusion of filaments in the yarn causes additional problems. During warp knitting fused filaments tend to cause yarn breaks, inferior warping and knitting performance and results in fabric of uneven appearance. Fused filaments also contribute to uneven dyeing and to a fabric having a stiffer and harsher hand.

An object of the present invention is to provide a nylon composition useful for producing antistatic yarn having fusion-free filaments.

Another object of the invention is to provide a fusion-free multifilament antistatic nylon yarn from said composition, using conventional equipment.

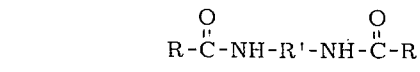
Still another object of the invention is to provide a simple and economical means for producing said yarn.

SUMMARY OF THE INVENTION

The present invention provides fusion-free multifilament, antistatic nylon yarn. This yarn is conventionally prepared on conventional nylon melt spinning equipment by extrusion of a nylon containing dispersed therein, based on the weight of nylon,

A. 1 to 20 percent of a polyalkoxylated triglyceride of a saturated fatty acid in which the polyalkoxy portion thereof has a molecular weight ranging from about 2200 to about 22,000 and wherein the saturated fatty acid has 10 to 30 carbon atoms, and

B. 0.3 to 2.0 percent of an alkylene bis-alkylcarboxamide of the formula



in which R is a C_{12} to C_{20} alkyl radical and R′ is a C_2 to C_4 divalent alkylene radical.

The antistatic yarn of this invention possesses good drawtwist performance and is readily processed into high quality fabrics without encountering the difficulties associated with yarns having fused filaments. Fabrics prepared from the yarn of the present invention are dyeable to uniform colors and have a pleasing appearance and hand.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

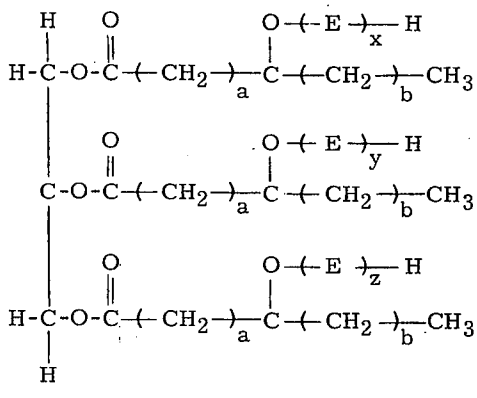
The nylon compositions and yarns of this invention comprise three components: nylon, triglyceride and alkylene bis-alkylcarboxamide.

Nylon Component

Nylons (i.e. polycarbonamides) particularly useful in preparing the fusion-free yarn of the present invention are of high molecular weight and prepared, for example, from either (a) substantially equimolar amounts of a diamine of the formula $NH_2(CH_2)_nNH_2$ and a dicarboxylic acid of the formula HOOC-R-COOH or (b) one or more amino acids or amide-forming derivatives thereof, where n is a number from 4 to 12 and R is $-(CH_2)_n-$ or phenylene. Homopolycarbonamides and copolycarbonamides of this type and their preparation are well known in the art and include: poly-ε-caprolactam (i.e. nylon 6), polyhexamethylene adipamide (i.e. nylon 66), polyhexamethylterephthalamide (i.e. nylon 6TA) and polydodecanylterephthalamide (i.e. nylon 12TA).

Triglyceride Component

Polyalkoxylated triglycerides of saturated fatty acids useful in practicing the invention are described in U.S. Pat. No. 3,388,104 and are of the formula



(formula I)

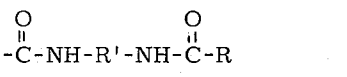
where *a* and *b* are integers between 2 and 26 with the proviso that *a*+*b* is at least 10, E is a C_2 to C_4 alkylene oxide radical and *x*, *y* and *z* are integers greater than zero the sum of which is a value between 50 and 500. A preferred triglyceride of this type is that represented by formula I when a is 10, *b* is 5, E is —CH_2CH_2O— and

x+y+z is 200, i.e. polyethoxylated hydrogenated castor oil, that is, polyethoxylated glycerol tristerate. This compound is conveniently referred to hereinafter as PHCO/200E.

It has been found that nylons containing between 2 and 8 percent on a weight basis of PHCO/200E possess exceptional antistatic properties. However, in using other triglycerides of formula I, greater or lesser amounts may be required depending on the degree of ethoxylation and on the molecular weight of the particular triglyceride.

Alkylene Bis-alkylcarboxamide Component

Preferred alkylene bis-alkylcarboxamides of the formula



(formula II) for use in practicing the present invention are the ethylene bis-alkylcarboxamides wherein the alkylcarboxamide groups each contain from 16 to 24 carbon atoms, for example, ethylene bis-stearamide.

It has been found that for any given nylon component containing from 1 to 20 percent by weight of the triglyceride component at least about 0.3 percent of the alkylene bis-alkylcarboxamide component, based on the weight of nylon, is required to provide fusion-free yarn and that the use of more than the amount of the carboxamide component required to provide fusion-free yarn does not significantly enhance the properties of the resulting yarn. It has also been found that when more than about 2.0 percent of the carboxamide component is present the fiber-forming qualities of the nylon composition deteriorates rapidly with increasing amounts of the carboxamide component. Accordingly, the nylon compositions of the present invention contain between 0.3 and 2.0 percent of the alkylene bis-alkylcarboxamide, based on the weight of nylon, and preferably contain only that amount of the carboxamide component required to provide fusion-free yarn.

Yarn Preparation

In the examples that follow nylon 66 textile yarn (40 denier, 13 filament) is melt-spun from nylon 66 flake on spinning machines or units that are conventionally used in commercial production of this yarn. In preparing the yarn nylon 66 flake is continuously fed to a melt chamber within the spinning machine where it is melted by means of a heated grid to form a melt pool. Molten polymer usually at a temperature between about 285°C. and 295°C. is then accurately metered from said melt chamber through filters and then orifices of a spinneret at which point molten filaments are formed. The molten filaments are cooled in an air chamber positioned vertically and immediately below the spinneret. After being solidified, the filaments are converged into a yarn bundle in the lower portion of this air chamber by any suitable means such as crossed pins. The convergence of the filaments is designed to take place at a distance approximately four feet below the spinneret. The resulting yarn bundle is heat treated, for example by passing it through an atmosphere of steam, and packaged, e.g. wound onto a bobbin.

The packaged yarn is thereafter subjected to a drawtwisting operation using conventional drawtwisters in which the yarn is stretched several times, twisted slightly and packaged for further processing into fabric.

The spinning machines used to prepare the yarn described in the examples were modified in that the melt chamber was provided with a conduit for adding the triglyceride component and/or other viscous liquids to the molten polymer just prior to filament formation and with a stirring means consisting of interleaving vanes on relatively rotating opposed discs for intimately blending added viscous liquids with the molten polymer. The modified melt chamber is shown and described in U.S. Pat. No. 3,603,563.

The alkylene bis-alkylcarboxamide component of the yarn may be incorporated therein by any suitable means, for example: the nylon flake that is fed to the spinning machine may be coated therewith; the carboxamide and triglyceride may be mixed, blended, extruded and chopped into pellets that are then mixed with the nylon flake; the carboxamide may be added to the nylon flake as the flake is being fed into the spinning machine; or the carboxamide may be added to the molten polymer and blended therewith.

Measurements and Tests

Fusion ratio (R_F) of a yarn is defined by the equation $R_F <= f_E$, wherein (f) is the number of filaments in the yarn and (E) is the number of free elements in the yarn where a free element is a single free filament or two or more fused filaments present in the yarn as a free bundle. Thus, a yarn composed of 13 filaments may have a fusion ratio (R_F) ranging from 1 (no fusion) to 13 (entire fusion), for example, if the yarn contains five free filaments, one free bundle composed of five filaments fused to one another and a second free bundle composed of three filaments fused to one another, this particular yarn has a fusion ratio (R_F) of 13/7 or about 1.86. Since filament fusion usually varies along the length of a multifilament yarn, it is desirable to determine an average (R_F) value for a given yarn. In the examples that follow an average (R_F) value is determined for each yarn by determining four (E) values at about 20 yard intervals along the length of each of four yarn samples. The resulting 16 (E) values are averaged and then an average (R_F) value calculated. (E) values are best determined visually after separating the yarn into its free elements. It is desirable that yarn used in manufacturing textile apparel fabrics have a fusion ratio of less than 1.20 and preferably a ratio of 1.00.

Static half-life of a fabric is determined by electrostatically charging the fabric and then measuring the time in seconds required for the decay or dissipation of one-half of the static charge built up on the fabric. Static testing of yarns described in the examples was accomplished on a Static Honestometer, available from Shishido and Co. Lmt. The Honestometer operates with a corona discharge as the source of electrical energy and with an oscilloscope as the detector and is described in Modern Textiles, pp. 68—70 (March, 1973).

In brief, the method of testing on the Honestometer involves attaching a suitable yarn sample (i.e. knit tube) to a turntable which is rotated at approximately 1750 revolutions per minute. A 10 KV discharge is impinged on the sample from a needle electrode placed about 20–25 mm. from the sample surface. An oscilloscope detector probe is located 180° across the turntable from the discharge (or driver) electrode and measures the charge leakage through a similar air gap from the sample to the probe on each revolution of the turn-

table. The charge build-up and decay on the sample are displayed as a series of pulses on the oscilloscope. Permanent records of the static properties of the sample are obtained by photographing the oscilloscope face. From the oscilloscope (or photographs) the initial charge build-up on the sample in millivolts (mv) and the static half-life of the sample are readily obtainable. All measurements are made at 70°F. and 30 percent relative humidity. Where additives are incorporated into a polymer to impart antistatic properties to yarns made therefrom, fabric samples of the resulting yarn are usually washed one or more times prior to testing to be certain that antistatic properties of the yarn will be permanent and not merely temporary.

A washing consists of agitating the fabric samples at 140°F. for a period of 20 minutes in a scour solution composed of 0.1 percent Triton X-100, which is a nonionic surfactant, and 0.1 percent tetrasodium pyrophosphate, said solution having a liquor-to-fiber ratio of 40:1. After washing the fabric samples are dried and conditioned for a period of 24 hours at 30 percent relative humidity and 70°F. temperature prior to testing.

In these tests, the shorter the time required for the dissipation of the half-life of the static charge build up on the fabric, the greater the degree of antistatic property. That is to say, the greater the antistatic properties of the fabric the less time required for the decay of half the static charge built up on the fabric.

The following examples further illustrate the invention. In the examples 40 denier, 13 filament nylon 66 yarn is melt spun from nylon 66 flake using the equipment and procedures described above. Percentages are by weight, based on the weight of nylon, unless otherwise indicated.

EXAMPLES 1-3

In these examples three yarns were prepared having the following compositions:

Example 1 Yarn - nylon 66

Example 2 Yarn - nylon 66 containing 5.5% PHCO/200E

Example 3 Yarn - nylon 66 containing 5.5% PHCO/200E and 1.4 percent ethylene bis-stearamide.

The ethylene bis-stearamide in this instance was added along with the PHCO/200E as a blend thereof to the melt chamber. Each of the yarns was examined and the fusion ratio (R_F) determined. Thereafter, each yarn was converted to a fabric sample, i.e. knit tubing, suitable for static testing. The static properties of each yarn was then determined as described herein. The fusion ratio and static properties of each yarn are given below. The yarn tubing of example 3 had a soft and pleasing hand, whereas the yarn tubing of example 2 had a stiffer and harsher hand.

FILAMENT FUSION

Yarn Sample	Average (R_F)
Ex. 1	1.0
Ex. 2	2.05
Ex. 3	1.0

STATIC PROPERTIES

Yarn Sample	No. of Washings	Charge Build-Up, mv	Static Half-Life, Sec.
Ex. 1	1	360	180
Ex. 2	1	78	24

FILAMENT FUSION-Continued

Yarn Sample		Average (R_F)	
Ex. 3	1	86	14
Ex. 2	10	94	109
Ex. 3	10	104	68

The foregoing examples illustrate that the nylon compositions of the present invention provide fusion-free multifilament antistatic yarns, whereas corresponding compositions differing only in that they do not contain an alkylene bis-alkylcarboxamide of the type described herein provide antistatic multifilament yarn having a high level of filament fusion.

EXAMPLE 4

Six 40 denier, 13 filament nylon yarns were melt-spun from molten nylon 66 containing 5.5% PHCO/200E and the indicated percentages of the following compounds:

A. 0.75 percent ethylene bis-stearamide
B. 0.75 percent aluminum tristearate
C. 0.75 percent sodium stearate
D. 0.75 percent microcrystalline wax
E. 1.00 percent stearamide
F. 1.25 percent oleamide.

The yarns were prepared in the same manner as the yarns of examples 1-3 with the exception that in preparing yarns A-E the indicated compound was added by coating the flake with the compound prior to feeding the flake to the spinning machine. The fusion ratios of these yarns were determined and are given below:

Yarn Sample	Fusion Ratio, Average (R_F)
A	1.07
B	1.48
C	2.74
D	4.34
E	3.24
F	2.69

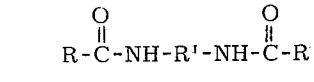
The above results show that only the yarn of present invention is composed of fusion-free filament. All the other yarns illustrated have an unacceptable level of filament fusion, i.e. a (R_F) greater than 1.20.

We claim:

1. A polycarbonamide composition suitable for melt spinning into multifilament fusion-free antistatic yarn, consisting essentially of a polycarbonamide having dispersed therein, based on the weight of polycarbonamide,

A. 1 to 20 percent of a polyalkoxylated triglyceride of a saturated fatty acid wherein the polyalkoxy portion thereof has a molecular weight ranging from about 2,200 to about 22,000 and wherein the saturated fatty acid has 10 to 30 carbon atoms, and

B. 0.3 to 2.0 percent of an alkylene bis-alkylcarboxamide of the formula



wherein R is a C_{12} to C_{20} alkyl radical and R' is a C_2 to C_4 alkylene radical.

2. The composition of claim **1** wherein the polycarbonamide is poly(hexamethylene adipamide).

3. The composition of claim 2 wherein said polyalkoxylated glyceride is polyethoxylated hydrogenated castor oil in which the ethoxylated portion thereof has a molecular weight of about 4,400.

4. The composition of claim 3 wherein said alkylene biscarboxamide is ethylene bis-stearamide.

5. The composition of claim 4 wherein said polyalkoxylated glyceride is present in an amount ranging from 2 to 8 percent.

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