

1

3,477,899

BIFILAR POLYAMIDE YARNS AND FILAMENTS
Harry Kubitzek, Otto Unger, Günter Nawrath, and
Alfred Reichle, Dormagen, and Günther Nischk, Lever-
kusen, Germany, assignors to Farbenfabriken Bayer
Aktiengesellschaft, Leverkusen, Germany, a corpora-
tion of Germany

No Drawing. Filed May 18, 1965, Ser. No. 456,827
Claims priority, application Germany, June 25, 1964,
F 43,260

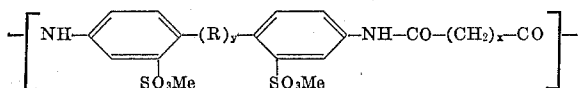
Int. Cl. D01d 5/10; D01f 3/28

U.S. Cl. 161—173

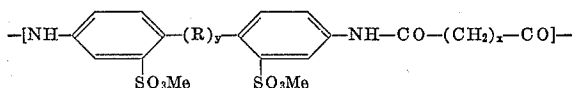
6 Claims

ABSTRACT OF THE DISCLOSURE

Polyamide bifilar yarns having a wool-like crimp comprising a simple synthetic linear polyamide component and a polyamide component containing 0.5 to 20% of the sulfonic acid moiety having the formula:



wherein R represents alkylene or alkene, y is a whole number of from 0 to 3, x is a whole number of from 2 to 12 and Me is hydrogen or an alkali metal; and a process of preparing such bifilar polyamide yarns having a wool-like crimp by melt spinning side-by-side, a simple synthetic linear polyamide together with a polyamide having 0.5 to 20% of the sulfonic acid moiety having the formula:



wherein R is alkylene or alkene, y is a whole number of from 0 to 3, x is a whole number of from 2 to 12, stretching the resulting bifilar yarn and subjecting the resultant stretched bifilar yarn to a heat treatment.

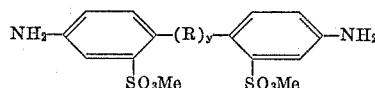
This invention relates to polyamide yarns, filaments, fibres and threads having a wool-like crimp and to a process for the production of these polyamide yarns.

It is known that polyamides of uniform composition, for example condensation products of ammonium salts of dicarboxylic acids with diamines or corresponding products obtained with ϵ -aminocaproic acids, and polymerization products of higher membered lactams spun on their own as monofilaments or multifilaments do not have any crimp in the threads or fibres even if they have been subjected to a heat treatment e.g. with hot air. A crimp can be imparted to these threads or fibres only by mechanical influences such as compression crimping. This type of crimping is not stable under severe mechanical stresses because once pulled out it is not reversible. It is also known that if polyamides of different compositions differing in their hydrophilic properties are spun together by bifilar spinning to form composite threads, the threads or fibres obtained have a crimp after the heat stretching and subsequent heat treatment in the relaxed state. This type of crimp occurs if, for example, polycaprolactam or the polyamide of hexamethylene diamine and adipic acid is spun by bifilar spinning with the polyamide of hexamethylene diamine and sebacic acid or a polyester to form a composite thread.

2

An object of this invention is to provide a process for preparing polyamide yarns, fibres and threads having a wool-like crimp in which polyamides containing aryl sulphonic acid groups are spun with other polyamides to form a composite bifilar thread. After stretching, heating in water or steam and drying, threads or fibres are obtained which assume a helical crimp, of wool-like character. By this process it is possible to spin a two-component filament composed of an only slightly modified polyamide and its unmodified counterpart in order to obtain crimped, wool-like threads or fibres.

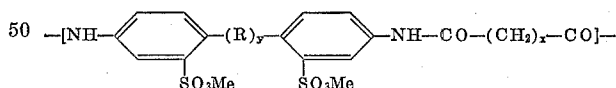
The polyamides containing aryl sulphonic acid groups are obtained by reacting 4:4'-diamino-2:2'-diphenyl-disulphonic acid or 4:4'-diamino-2:2'-diphenyl-alkanedisulphonic acids of the formula



in which R denotes a CH_2 group or an alkene group which may be substituted, y denotes the numbers 0 to 3 and Me denotes hydrogen or an alkali metal, with approximately equivalent quantities of alkylene dicarboxylic acids or their alkali metal salts in quantities of up to 20% by weight of polyamide-forming starting materials such as lactams or salts of diamines and dicarboxylic acids, and spinning the resulting polyamides from the melt to form fibres or threads.

Examples of diaminosulphonic acids suitable for the process of the invention are 4:4'-diamino-2:2'-diphenyl-disulphonic acid, 4:4'-diamino-2:2'-dibenzyl-disulphonic acid, 4:4'-diamino-2:2'-diphenyl-propanedisulphonic acid. These compounds are present as internal ammonium salts. In the polycondensation of the polyamide-forming starting materials, these internal ammonium salts are either used together with dialkali metal salts of dicarboxylic acids or the internal ammonium salts of sulphonic acid are converted into the corresponding alkali metal salts and condensed in the presence of the free dicarboxylic acid. Examples of dicarboxylic acids or their corresponding alkali metal salts are succinic acid, adipic acid, suberic acid and sebacic acid. To obtain high polymer polyamides suitable for spinning, the diamines containing sulpho groups should preferably be used with the equivalent quantity of dicarboxylic acid or its alkali metal salts.

In a particular method of carrying out the invention, 0.5 to 20%, preferably 1.5 to 10% of the following structural element:



wherein R denotes a CH_2 group or an alkene group which may be substituted, y denotes the numbers 0-3, x denotes the whole number from 2 to 12 and Me denotes hydrogen or an alkali metal, should be contained in the polyamide chain.

It is, of course, also possible to spin a suitably modified type of polyamide with a non-modified polyamide of a different composition to form a bifilar thread. Examples of suitable combinations include the polyamide of hexamethylenediamine and sebacic acid spun with the modified polyamide of caprolactam, which polyamide contains sulphonic acid groups.

The bifilar compound thread is obtained by separately melting the resulting modified polyamide components and the non-modified polyamides and spinning the two together

3

at temperatures between 250 to 300° C., preferably 265 to 285° C., in such a manner that the two components adhere together over the whole length of the threads.

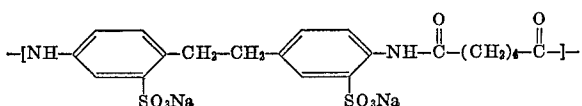
The resulting bifilar compound threads are then stretched at room or elevated temperature. The stretching temperature depends on the composition of the polyamides and is normally between 20 and 140° C. The threads are then subjected to a heat treatment with water at temperatures of about 60 to 120° C., the presence of a crimp in the threads being indicated even while the threads are still moist by the different shrinkage on boiling. The heat treatment may be carried out with hot water or steam. The intensity of the crimp already indicated depends not only on the shrinkage on boiling but also on the difference between the hydrophilic properties of the two polyamides in the spun bifilar. During the drying process, the hydrophilic part of the bifilar thread shrinks to a much greater extent so that an extremely intense and uniform three-dimensional crimp is obtained in this way and a wool-like character is imparted to the product. If these threads are placed in cold or warm water, the more hydrophilic part of the bifilar thread takes up considerably more moisture than the hydrophobic part, the crimp being thereby caused to disappear to a greater or less extent, but the thread reverts almost completely to its original crimped state when dried again. This process can be repeated as often as desired without any loss in the original crimp effect, not even any loss in the intensity of the effect. Even if the shrinkage on boiling is eliminated by excessive overstretching, the original crimp can be completely recovered on heating in warm water, i.e. the crimped effect is in all cases reversible.

Hot air produces a crimp if the stretched bifilar threads are heat treated, but this crimp is not removed by treatment with water. If threads treated in this way are in addition introduced into hot water, the reversible crimp released by hot water is added to the crimp already present. In addition, the bifilar fibres produced by this method can very easily be dyed with basic dyestuffs and are themselves very fast to light.

The following examples illustrate more specifically the invention and describe specific embodiments of the invention.

EXAMPLE 1

Unmodified poly (ϵ -aminocaproic acid) whose 1% solution in m-cresol had a relative solution viscosity of 2.7 at 25° C. and a poly (ϵ -aminocaproic acid) modified with 5% of the structural unit;



and having the same relative solution viscosity were spun at 280° C., with an air temperature of 25° C., to form bifilar compound threads. The spinning pumps were adjusted so that the proportion by weight of the quantities of unmodified and modified components discharged was 1:1. Employing a draw-off speed of 800 m./min. a multifilar thread was drawn off a 12 aperture spinneret having apertures of 0.25 mm. in diameter. This thread was stretched in the ratio of 1:3.5 between two plates heated to 80° C. When treated with hot water at 90° C. and dried in the relaxed state, this thread developed an intensive spiral crimp.

EXAMPLE 2

A multifilar compound thread was produced as in Example 1, the proportions of the two components discharged being 1 part by weight of unmodified and 2 parts by weight of modified poly (ϵ -aminocaproic acid).

EXAMPLE 3

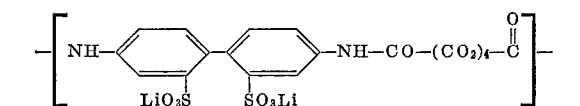
Bifilar compound threads were spun from the components according to Example 1, at a spinning temperature of 280° C. and a rate of 400 m./min. After being stretched

4

ing the ratio of 1:4 between plates heated to 20 to 180° C., the compound threads had individual titres of 15 den. The two components were discharged in the proportion of 1:1. The spiral crimp developed according to Example 1 had a lower crimp number (number of convolutions per cm.) than the 5 den thread of Example 1, and a larger diameter of the crimp spiral.

EXAMPLE 4

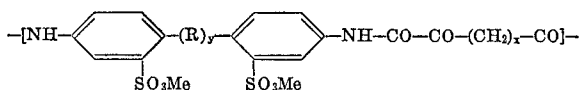
Unmodified polycaprolactam having a relative viscosity of 2.7 and a polycaprolactam modified with 5% of the structural unit



and having the same relative viscosity was spun together in air at 280° C. to form a compound thread. The proportion by weight of the quantities of modified and unmodified components discharged was 1:1. A multifilar thread was drawn off at the rate of 600 m./min. from a 12 aperture spinneret having an aperture diameter of 0.25 mm., and this thread was stretched in the ratio of 1:3.5 between two plates heated to 80° C. When treated with hot water at 95° C. and dried in the relaxed state this thread develops an intensive crimp.

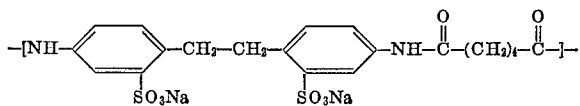
We claim:

1. A bifilar yarn having a wool-like crimp consisting essentially of (1) a polyamide component selected from the group consisting of polycaprolactam and polyaminocaproic acid and (2) a modified polyamide component selected from the group consisting of modified polycaprolactam and modified polyaminocaproic acid containing as the modifying moiety 0.5 to 20% based on the polyamide forming starting materials, of the moiety having the formula:

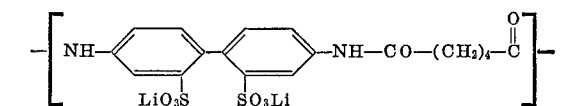


wherein R is a member of the group consisting of alkylene and alkene, y is a whole number of from 0 to 3, x is a whole number of from 2 to 12 and Me is a member of the group consisting of hydrogen and an alkali metal.

2. A bifilar yarn according to claim 1 wherein said sulfonic acid group moiety has the formula:



3. A bifilar yarn according to claim 1 wherein said sulfonic acid group moiety has the formula:



4. A bifilar yarn according to claim 1 wherein the proportion by weight of said polyamide component containing the sulfonic acid group moiety (2) to said polyamide component (1) is in the range of from about 10:90 to 90:10.

5. A bifilar yarn according to claim 1 wherein the proportion by weight of said polyamide component containing the sulfonic acid group moiety (2) to said polyamide component (1) is in the range of from about 30:70 to 70:30.

6. A bifilar yarn according to claim 1 wherein said polyamide component (2) contains from 1.5 to 10% based on the polyamide forming starting materials of said sulfonic acid group moiety.

(References on following page)

5

References Cited

UNITED STATES PATENTS

2,989,798 6/1961 Bannerman.
 3,039,990 6/1962 Hoffman ----- 260—857
 3,182,100 5/1965 Bedell.
 3,216,965 11/1965 Cipriani ----- 260—857

5

6

3,225,534 12/1965 Knospe.
 3,328,484 6/1967 Lugaz et al. ----- 260—857

HAROLD D. ANDERSON, Primary Examiner

U.S. Cl. X.R.

57—140; 161—177; 260—78, 857; 264—171, 210

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,477,899 Dated Nov. 11, 1969

Inventor(s) HARRY KUBITZEK, OTTO UNGER, GÜNTER NAWRATH and
ALFRED REICHLE

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 1, line 43: "relate" should be -- relates --

Claim 1, in the right-hand portion of the structural formula:

"NH-CO-CO-(CH₂)_x - CO]- " should be -- NH-CO-(CH₂)_x-CO] --

SIGNED AND
SEALED
APR 28 1970

(SEAL)

Attest:

Edward M. Fletcher, Jr.
Attesting Officer

WILLIAM E. SCHUYLER, JR.
Commissioner of Patents