MANUFACTURE OF FILAMENTS OR FIBERS OF POLYAMIDES

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No Drawing. Filed Sept. 19, 1972, Ser. No. 290,321

Claims priority, application Germany, Sept. 20, 1971, P 21 46 858.9

Int. Cl. C08g 41/04

U.S. Cl. 260—857

4 Claims

ABSTRACT OF THE DISCLOSURE

Manufacture of filaments or fibers of synthetic linear polyamides containing from 2 to 15% by weight of alkylated polyamides using certain conditions concerning the fineness of subdivision, filtration and shaping.

This invention relates to a process for the manufacture of filaments or fibers containing alkylated polyamides. It is well known that textiles and carpets of synthetic fibers including polyamides build up an electrostatic charge due to friction occurring in use. Such electrostatic charges, in the case of textiles, may produce an unpleasant feel when said textiles are in contact with the human body, or, in the case of carpets, persons walking thereon may receive highly unpleasant shocks due to electrostatic discharges. These phenomena are particularly observed when the relative humidity is low.

There has been no lack of attempts to obviate these undesirable properties. For example, attempts have been made to increase the surface conductivity of polyamide filaments by applying antistatic agents thereto, either together with the lubricant or in a subsequent step. Such agents are usually water-soluble, however, and, consequently their effectiveness disappears on washing. The effect may be made more permanent by incorporating the agents in the polymer. This method is described in U.S. Pat. No. 3,575,577, which teaches the incorporation of polyethylene glycols into polyamide filaments. However, the results obtained could still be improved upon, since the said polyethylene glycols are extracted on repeated washing. These well-known disadvantages may, in theory, be overcome by subsequent cross-linking with, say, X-rays, but this solution is hardly economical.

It is an object of the invention to manufacture filaments or fibers which show a permanent greatly reduced tendency to antistatic build-up without the other properties of the fibers, such as their mechanical or textural properties, being substantially impaired.

This object is achieved by a method of making filaments or fibers of filament-forming synthetic linear polyamides containing alkylated polyamides in an amount of from 2 to 15%, on the weight of the filaments or fibers, wherein in a molten mixture of the filament-forming synthetic linear polyamides and alkylated polyamides is subjected to thorough mixing so that the alkylated polyamides are present in the finished filaments in the form of particles having a diameter of not more than 0.1 μ, and the molten mixture is filtered at least once through a filter having a pores width of not more than 20 μ, the mixture is converted to chip form if desired, and the mixture is melt spun, the molten mixture being maintained at temperatures of up to not more than 290° C. for a total period of not more than 30 minutes.

By filament-forming synthetic linear polyamides of high molecular weight we mean those having recurring amide groups in the backbone.

Suitable filament-forming or fiber-forming synthetic linear polyamides are, for example, the polycondensates of lactams having from 4 to 12 carbon atoms, such as e-caprolactam, octolactam, dodecanolactam or mixtures of said lactams, and the polycondensates of salts of diamines and dicarboxylic acids having from 4 to 12 carbon atoms, such as the salts of adipic acid, suberic acid or sebacic acid, and hexamethylenediamine, octamethylene diamine or dodecamethylene diamine or copolyamides of said polyamides and polyamides containing alkyl groups, which may be linear or branched.

The amount of alkylated polyamides used is conveniently from 2 to 15% and in particular from 4 to 10% w/w of the weight of the filaments or fibers. It is possible to use larger proportions, but this leads to changes in the properties of the filaments or fibers.

The solid components, i.e. the filament-forming polyamides and the alkylated polyamides, may be melted separately or together. The components may be mixed in a conventional single-worm or twin-worm extruder, of which one or both of the worms may be axially displaceable if desired. Static mixers having stationary conducting elements may also be advantageously used. Adequate mixing is usually obtained using static mixers having 15 or more elements. Static mixers of this kind are described for example in U.S. Patents 3,286,992 and 3,404,689 and German Published Application 1,788,335.

The amount of blended polyamides is conveniently from 2 to 15% and in particular from 4 to 10% w/w of the weight of the filaments or fibers. It is possible to use larger proportions, but this leads to changes in the properties of the filaments or fibers.

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Conditions should be such as to achieve such thorough mixing that the alkylated polyamides, which form a separate phase in the filaments after cooling and drawing, have a particle diameter of not more than 1 μ. The degree of mixing required to achieve this end may be previously found by simple experiment. The length of the separate particles of alkylated polyamides in the filaments after drawing is generally a multiple of their diameter, for example about 1μ or more. It is an important feature of the process of the invention that filtration of the molten mixture of components is carried out at least once through filters hav-
ing pore widths or not more than 20 μ. Examples of suitable filters are metal sieves or, in particular, sintered metal filters such as sintered metal plates or sintered metal tubules.

Following the thorough mixing and filtration, the molten mixture may, if desired, be converted to chips and subsequently melt-spun to filaments or fibers. Alternatively, the molten mixture may be thoroughly mixed and filtered and immediately melt-spun to filamentary fibers without intermediate conversion to chip form. Another alternative is to carry out mixing and filtration in the spinning apparatus by using a spinning extruder capable of providing thorough mixing and placing a filter having a pore width of not more than 20 μ immediately downstream of the spinning nozzle, which filter is advantageously an appropriate sintered metal filter.

It is important that the molten mixture of filament-forming polyamide and alkoxylated polyamide is maintained at temperatures of not more than 200 °C and generally at from 250 to 260 °C, a period of not more than 30 minutes including the melt-spinning time. The time during which the mixture may be in chip form in a colder state is, of course, not included.

The filaments are made using conventional spinning systems. In course, the filaments produced are also observed in the manufacture of polyamide materials, such as the exclusion of oxygen and the use of certain low moisture contents, must be met. The filaments or fibers produced by melt-spinning may have a round cross-section or a profiled cross-section such as a trilobate or tetralobate cross-section. The filaments may be in the form of two-component filaments with other filament-forming materials. Conveniently, the filaments or fibers of the invention are stretched, for example at a draw ratio of from 1:2.5 to 1:4.5. The filaments or fibers may also be texturized, for example by the false-twist or stuffer-box method or by texturizing in moving gas media.

The filaments or fibers may incorporate conventional additives such as pigments, for example titanium dioxide, or light stabilizers, heat stabilizers, and stabilizers to prevent the degradation of polymer chains.

The filaments or fibers produced by the method of the invention are distinguished by their greatly reduced tendency to build up electrostatic charges. This good anti-static behavior remains after repeated washing.


It is a great advantage that the good anti-static properties of the filaments or fibers produced by the method of the invention are not obtained at the expense of the mechanical and textural properties of the filaments or fibers. The process of the invention is distinguished in that the yields produced in spinning and drawing are high as compared to unmodified polyamide, which means that the number of defects found in the subsequent stages of warping and knitting is low. The same applies to the use of the filaments in the manufacture of carpets, where great advantage are found, for example, in the steps of texturizing and tufting. The quality of the filaments is tested by its standard technological, mechanical properties and dyeability. The values found for the filaments produced by the invention hardly differ from those provided by the corresponding unmodified polyamides. Indeed, the filaments modified by the present invention can usually be dyed deeper and more level shades— a frequently desirable feature. Since the streak-free dying of conventional polyamides remains a problem, the above improvement as concerns dyeableness is particularly advantageous.

The filaments made in accordance with the present invention may be woven or knitted to textile articles such as underwear and shirts with advantageous results. The quality required in such articles is particularly high and the measures of the invention are therefore especially effective for this application. Filaments made in the manner proposed by the present invention may also be used successfully in carpets, both in the form of continuous filaments and as staple fibers. A distinct improvement in the quality of the filaments is achieved as compared with prior art modifications for the manufacture of two-phase antistatic filaments.

In the following Example the parts and percentages are by weight. The relative viscosities (ηspel) are measured on a 1% w/w. solution in 98% H₂SO₄ at 20 °C.

**EXAMPLE**

A Werner & Pfleiderer twin-twin extruder ZSK 53 is fed with 92.5 parts of extracted and dried polycaprolactam chips having a relative viscosity (ηspel) of 2.4 and delustered with 1.6% of TiO₂ and simultaneously with 7.5 parts of an ethoxylated polycaprolactam showing a ratio of polycaprolactam to ethylene oxide of 1:3.6 by weight. Heating is effected so that the temperature of the molten mixture rises from 220 °C. at the feed end to from 268 °C to 268 °C at the output end of the extruder. At a speed of 100 in. per minute, the throughput is 80 kg/hr. The residence time of the melt in the extruder is about 2 minutes.

At the end of the screws there is provided a filter unit which is heated at 250 °C. and is fitted with metal sieves of 17 μ mesh. The filtered molten mixture is extruded in four strands which are cooled in a water bath and then granulated. The granules are dried to a water content of less than 0.1% and then melt-spun to filaments 14 f 10 dtex. using an extruder provided with a sintered metal fiber having an average pore width of about 16 μ. The residence time of the molten mixture in the spinning system is about 15 minutes. The resulting filaments are then stretched at a draw ratio of 1:3.28 to form filaments 4 f 10 dtex. The drawing yield is 96% based on cops weighing 1 kg.

Table 1 below gives the results of tests in comparison with unmodified polycaprolactam filaments:

<p>| Table 1 |</p>
<table>
<thead>
<tr>
<th>Filaments of invention</th>
<th>Filaments of unmodified polycaprolactam</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total denter (dtex)</td>
<td>64.4</td>
</tr>
<tr>
<td>Number of capillaries</td>
<td>16</td>
</tr>
<tr>
<td>Capillary denter (dtex)</td>
<td>4.45</td>
</tr>
<tr>
<td>Tenacity (dry g/m²)</td>
<td>195</td>
</tr>
<tr>
<td>Breaking length (kilocentimeters)</td>
<td>63.0</td>
</tr>
<tr>
<td>Elongation (dry) (percent)</td>
<td>30.7</td>
</tr>
<tr>
<td>Loop tenacity, absolute (kilocentimeters)</td>
<td>85.2</td>
</tr>
<tr>
<td>Loop tenacity, relative (percent)</td>
<td>86.3</td>
</tr>
<tr>
<td>Twist (turns/meter)</td>
<td>13.2</td>
</tr>
<tr>
<td>Shrinkage on bolling (percent)</td>
<td>-13.7</td>
</tr>
</tbody>
</table>

This table shows that there are no substantial differences between the two types of filaments. The ethoxylated polycaprolactam is present in the filaments as a separate phase in the form of very small rods having a diameter of not more than 0.1μ. Using the apparatus described by Hoyl & Littgen (loc. cit.) for measuring the electrostatic build-up, knitted material made from the filaments of the invention and which has been washed five times has a field strength of only 60 volts/cm, when measured at 50% relative humidity after being rubbed for 4 minutes against a knitted fabric of unmodified polycaprolactam. Unmodified polycaprolactam in the form of knitted fabric which has been washed five times shows a field strength of 2,400 volts/cm, when measured under similar conditions.

Knitted fabric made from the filaments of the invention shows very uniform affinity for acid dyes as well as disperse dyes and also shows a distinctly higher color yield than unmodified polycaprolactam material.
We claim:

1. A process for the manufacture of antistatic polyamide filaments which comprises:
   forming a molten mixture of
   (a) a filament-forming synthetic linear aliphatic polyamide having recurring amide groups in the
   backbone and having a relative viscosity in the range of 1.5 to 2.9, measured as a 1% w/w.
   solution in 98% H₂SO₄ at 20° C., together with
   (b) from 2 to 15% by weight, based on the filaments or fibers, of an alkoxyalted polyamide
   manufactured by reaction of a polyamide selected from the group consisting of the poly-
   condensates of lactams of 4 to 12 carbon atoms and polycondensates of the salts of diamines
   and dicarboxylic acids of 4 to 12 carbon atoms with a 1,2-alkylene oxide in a weight ratio of
   from 1:2 to 1:5 and to provide a total molecular weight of 10,000 to 80,000,
   components (a) and (b) being so thoroughly mixed that said alkoxyalted polyamide is present in the
   finished filaments in the form of particles having a diameter of not more than 0.1 μ; and
   filtering said molten mixture at least once through a
   filter having a pore width of not more than 20 μ; and
   subsequently melt spinning said mixture into solid fila-
   ments, the molten mixture being maintained in this
   process at elevated temperatures of up to not more
   than 290° C. for a total period of not more than 30
   minutes.

2. A process as claimed in Claim 1, wherein the fila-
   ment-forming linear polyamide (a) is thoroughly mixed
   with from 4 to 10% by weight of alkoxyalted polyamide
   (b).

3. A process as claimed in Claim 1 wherein the mix-
   ture is converted into chip form or granulated interme-
   diate the filtering step and the melt spinning step.

4. The filamentary product obtained by the process of
   Claim 1.

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JAY H. WOO, Primary Examiner

U.S. Cl. X.R.

260—78 SC; 264—176 F