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PROCESS FOR PROVIDING POLYAMIDE FIBERS OF RELATIVELY LOW SOIL RETENTIVENESS

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No Drawing. Continuation-in-part of abandoned application Ser. No. 568,769, July 29, 1966. This application Sept. 22, 1969, Ser. No. 860,055
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ABSTRACT OF THE DISCLOSURE

Polyamide fibers of relatively low soil retentiveness are provided by drawing the fibers while coated with a liquid substance selected from the group consisting of glycerol, at least one C_2–C_6 alkylene glycol, at least one poly(C_2–C_4 alkylene glycol) having a molecular weight between 106 and about 600 and mixtures thereof over a drawing surface heated to a temperature between 130° and 220° C.

REFERENCE TO RELATED APPLICATION

This application is a continuation-in-part of my copending application Ser. No. 568,769 which was filed on July 29, 1966 and now abandoned.

BACKGROUND OF THE INVENTION

It is well known that among the properties normally considered in the selection of synthetic fibers for use in textile products such as carpets and apparel is the soil retentiveness of the fibers, i.e., the degree to which soil is not removed from the fibers by conventional cleaning techniques such as vacuuming, laundering, etc. Although polyamide fibers are generally satisfactory in that regard, a process by which their soil retentiveness can be lowered still further is very desirable and, accordingly, it is an object of this invention to provide such a process and the resulting polyamide fibers characterized by relatively low soil retentiveness.

SUMMARY OF THE INVENTION

It has now been found that the aforementioned objectives can be achieved by a process which comprises drawing polyamide fibers coated with at least about one percent, based on the weight of the fibers, of a liquid substance selected from the group consisting of glycerol, at least one C_2–C_6 alkylene glycol, at least one poly(C_2–C_4 alkylene glycol) having a molecular weight between 106 and about 600 and mixtures thereof over a drawing surface heated to a temperature between about 130° and about 220° C.

DETAILED DESCRIPTION OF THE INVENTION

The polyamides of which the fibers treated by the process of this invention are composed are the synthetic linear polyamides prepared by condensation polymerization procedures well known in the art or by other methods which do not involve a polycondensation reaction. Such polyamides are long-chain, high molecular weight polymers having recurring amide groups as an integral part of the polymer chain. Specific examples include polyhexamethylene adipamide, polycaproamide and polyhexamethylene sebacamide. Many other examples which include homopolyamides, copolyamides, terpolyamides, etc. are described in the technical and patent literature (e.g. in U.S. Pat. Nos. 2,071,253 and 2,130,948). Such polyamides are generally formed into fibers directly from the molten state and the fibers so formed are normally drawn in the solid state to increase their molecular orientation along the fiber axis and thereby increase the tenacity of the fibers. The drawing is conventionally carried out by advancing the fibers over one or more rotating feed rolls and then over one or more draw rolls rotating at a peripheral speed between about 2.5 and about 6 times (with most polyamides, between about 3.5 and about 5 times) the peripheral speed of the feed rolls after which the drawn fibers are taken up in an orderly fashion. If desired, the site of drawing can be localized by passing the fibers in frictional contact with a drawing surface (e.g. a draw pin or shoe) between the feed roll and draw roll and the use of such an intermediate drawing surface is preferred in the practice of this invention although, in the absence thereof, the draw roll or rolls may serve as the drawing surface of the present process.

Most polyamide fibers for which soil retentiveness is an important property are drawn in a single stage and the process of this invention is most advantageously applied to polyamide fibers during such single-stage drawing, i.e., to polyamide fibers that are substantially undrawn. However, it should be understood that the soil retentiveness of the fibers can be likewise substantially lowered by application of the present process to coated polyamide fibers which have been previously partially drawn (as in a multi-stage drawing procedure) and, hence, the process of this invention is also satisfactorily carried out at substantial draw ratios of less than about 2.5 (e.g. about 1.5) and applicable to polyamide fibers which are not substantially undrawn.

As aforesaid, the process of this invention is carried out by drawing polyamide fibers having coated thereon a liquid substance selected from the group consisting of glycerol, at least one C_2–C_6 alkylene glycol, at least one poly(C_2–C_4 alkylene glycol) having a molecular weight between 106 and about 600 and mixtures thereof. α,ω-Alkylene glycols and poly(α,ω-alkylene glycol)s are preferred although other alkylene glycols and poly(alkylene glycol)s are also suitable in most cases. The liquid substance can be coated on the fibers advancing toward the drawing surface by any convenient technique, e.g. by passing the fibers over a rotating liquid applicator roll in rubbing contact with a sponge to which the liquid substance is continuously supplied or through a bath of the liquid in which the drawing surface may be submerged, if desired.

The drawing surface temperatures that are useful in the process of this invention are those between about 130° and about 220° C. and which, together with the time of contact between the fibers and the drawing surface, are sufficient to provide a substantial lowering of the soil retentiveness of the fibers without substantial breakage, fusing or embrittlement of the fibers. Relatively lower drawing surface temperatures can be employed with the liquid substances of relatively lower molecular weight or carbon atom content as can be seen from the following table in which various examples of the liquid substances suitable for use in the process of this invention are listed together with the ranges of drawing surface temperatures which can be used to provide, with each such substance, a substantial lowering of the soil retentiveness of the polyamide fibers without substantial breakage, fusing or embrittlement of the fibers, i.e., without such breakage, fusing or embrittlement as would render the fibers unsuitable for use in the production of a polyamide fiber-containing product.

Liquid substance:	Drawing surface temperature, ° C. (approx.)
Glycerol (1,2,3-propane triol)	130°–180°
Ethylene glycol (1,2-ethane diol)	130°–165°
Propylene glycol (1,3-propane diol)	135°–175°
Butylene glycol (1,4-butane diol)	140°–185°
Pentylene glycol (1,5-pentane diol)	145°–195°
Hexylene glycol (1,6-hexane diol)	150°–205°
Diethylene glycol (MN=106)	130°–190°
Triethylene glycol (MN=150)	137°–201°
Tetraethylene glycol (MW=194)	143°–212°
Hexaethylene glycol (MW=282)	157°–220°
Polyethylene glycol (MW=400)	175°–220°
Polyethylene glycol (MW=600)	175°–220°
Dipropylene glycol (MW=134)	134°–197°
Tripropylene glycol (MW=192)	143°–212°
Tetrapropylene glycol (MW=250)	152°–220°
Pentapropylene glycol (MW=308)	161°–220°
Polypropylene glycol (MW=400)	175°–220°
Dibutylene glycol (MW=162)	139°–204°
Tributylene glycol (MW=234)	149°–220°
Tetrabutylene glycol (MW=306)	160°–220°

As shown by the foregoing table, the ranges of drawing surface temperatures that are useful in the process of this invention may be expressed as follows: when the liquid substances is glycerol, the temperature is between about 130° and about 180° C.; when the liquid substance is a C_2–C_6 alkylene glycol, the temperature is between about $(120+5n)$° and about $(145+10n)$° C. wherein n represents the number of carbon atoms in the alkylene glycol; when the liquid substance is a poly(C_2–C_4 alkylene glycol) having a molecular weight between 106 and about 400 such as diethylene glycol, tripropylene glycol, etc., the temperature is between about $(114.3+0.15\ MW)$° and about $(163.5+0.25\ MW)$° C. wherein MW represents the molecular weight of the poly(alkylene glycol); and when the liquid substance is a poly(C_2–C_4 alkylene glycol) having a molecular weight between about 400 and about 600, the temperature is between about 175° and about 220° C. When the liquid substance is a mixture of two or more of the aforementioned compounds, the minimum drawing surface temperature may be approximately as low as the lowest of the minimum temperatures that would be useful with the individual mixture components and the maximum drawing surface temperature may be approximately as high as the highest of the maximum temperatures that would be useful with the individual mixture components, depending on the composition of the mixture.

In most commercial operations, the time of contact between the fibers and the drawing surface will be generally between about 0.005 and about 0.5 second and most desirably between about 0.01 and 0.25 second, although the process of this invention can be carried out with satisfactory results using contact times that are shorter or considerably longer than those within the former of the foregoing ranges. Relatively shorter times within those ranges will be generally more desirable with relatively higher drawing surface temperatures and when the fibers are coated with one of the aforementioned liquid substances of relatively low molecular weight or carbon atom content. When the fibers are coated with the liquid substance a substantial length of time before contact with the heated drawing surface, superior results (e.g. greater ease of drawing) are generally obtained by maintaining the temperature of the coated fibers substantially below the minimum drawing surface temperature that would be useful with the same liquid substance in the process of this invention and preferably not higher than 100° C. until less than about 3 seconds and preferably less than about 1 second before the contact between the fibers and the drawing surface.

Substantial lowering of the soil retentiveness of the polyamide fibers can be achieved by the process of this

invention when the coating of the aforedescribed liquid substance on the fibers is present in any amount of at least about one percent, based on the weight of the fibers. Although superior results are generally obtained when the coating contains between about 2.5 and about 20 percent of the liquid substance, based on the weight of the fibers, it should be understood that the process can also be satisfactorily carried out when the coating contains higher percentages of the liquid substance such as, for example, when the drawing surface is submerged in a bath of the liquid substance. The liquid substance may be present on the fibers in a substantially pure form or it may be present in a coating containing any other solid or liquid additive or finish (e.g. a lubricant or antistatic agent) desirably present on the fibers during drawing. Although a substantially uniform coating of the liquid substance is generally preferred, good results are also obtained by the process of this invention when the coating of the liquid substance on the fibers is not substantially uniform.

In the following examples, the soil retentiveness of the drawn polyamide fibers is measured and expressed as the loss of brightness (tristimulus) value which results when textile products made from the fibers are solided and then vacuum-cleaned under carefully controlled conditions. However, there are other equally satisfactory methods of measuring the degree to which soil is not removed from polyamide fiber-containing products by conventional cleaning techniques and it will be apparent that the applicability of the process of this invention is not limited to polyamide fibers intended for use in any particular type of product, in products to be cleaned of soil by any particular method or in products for which the soil retentiveness of the fibers can be measured only by measurement of such brightness values.

EXAMPLE I—ETHYLENE GLYCOL

Substantially undrawn polyhexamethylene adipamide fibers consisting of six filaments having a total denier of 465 were drawn at a draw ratio of 4.54 over a ¾-inch diameter draw pin immersed in a bath of liquid ethylene glycol maintained at about 135° C. and then taken up at a speed of 118 feet per minute. The fibers were in contact with the ethylene glycol in the bath for about 0.38 second prior to contact with the pin, for about 0.2 second during contact with the pin and for about 0.13 second after contact with the pin. Seven-inch lengths of circular (2.5-inch diameter) tubing knit from the drawn fibers were blank-dyed for 30–60 minutes at 80–90° C. in 200 milliliters per five grams of tubing of an aqueous solution containing 0.1 percent by weight of isooctyl phenyl polyethoxy ethanol and 0.1 percent by weight of tetrasodium pyrophosphate and then washed five times with at least 100 milliliters of distilled water per five grams of tubing. After drying in room-temperature air for 16 hours, the tubing samples had an average brightness (Y) value of 77.7 as measured by a General Electric spectrophotometer. Each tubing sample was thereafter soiled by shaking it for two minutes in a paper bag containing two grams of a typical carpet soil having particle diameters not larger than 75 microns and then thoroughly vacuum-cleaned while flattened between ten-mesh screens. The vacuuming was carried out by slow, overlapping strokes with the open end of a piece of 17-millimeter diameter tubing attached to a vacuum source of 50 mm. Hg pressure. Both sides of each sample were completely vacuumed twice, once with lengthwise strokes and once with widewise strokes. As measured with the same spectrophotometer and filter, the average brightness value of the soiled and vacuum-cleaned tubing samples was 40.8, indicating that the retention of soil by the tubing samples had resulted in an average brightness loss of 47.6%.

COMPARATIVE EXAMPLE A

When the procedure of Example I was repeated with the exception that the application of ethylene glycol was omitted and the draw pin was otherwise maintained at about 135° C., the average brightness loss due to retention of soil by the tubing samples was 60.4%.

EXAMPLE II—ETHYLENE GLYCOL

Substantially undrawn polyhexamethylene adipamide fibers coated with about 1% of a commercial spin-finish, containing about 0.18% by weight of titanium dioxide and consisting of 68 filaments having a total denier of 4260 were drawn at a draw ratio of 3.69 over a 1.5-inch diameter draw pin maintained at about 150° C. and then textured and taken up at a speed of 1240 feet per minute. The fibers were in contact with the draw pin for about 0.06 second. Immediately before being drawn, the fibers were substantially uniformly coated with about 10%, based on the weight of the fibers, of liquid ethylene glycol by contact with an applicator roll to which the ethylene glycol was metered at room temperature. After being drawn and textured, the fibers were texturized with mechanical crimping gears and then tufted into carpet having a polyurethane backing. Small samples of the carpet were blank-dyed by boiling them for one hour in 40 grams per gram of sample of an aqueous solution containing ½% of Igepon T–33, based on the weight of the samples, and then rinsed for 20 minutes in 20 grams of water per gram of sample. After drying in room-temperature air for at least 40 hours, the carpet samples had an average brightness (Y) value of 71 as measured by a Photovolt photoelectric reflection meter (Model 610). Each sample was thereafter soiled by tumbling it for two minutes at one r.p.m. in a one-gallon jar containing one gram per ten grams of carpet sample of a synthetic soil containing, by weight, 52.5% sifted sawdust, 22.3% sifted peat moss, 10.9% calcium carbonate, 6.6% animal charcoal powder, 2.1% silica, 2.1% cement, 2.1% kaolin clay, 1.1% mineral oil, 0.2 furnace black and 0.1% red iron oxide. Each sample was then thoroughly vacuum-cleaned with slow overlapping strokes, three times lengthwise and three times widthwise, using a Sears, Roebuck hand vacuum cleaner having a ¾-inch diameter intake nozzle. As measured with the same reflection meter, the average brightness value of the soiled and vacuum-cleaned carpet samples was 45, indicating that the retention of soil by the carpet samples had resulted in an average brightness loss of 36.6%.

COMPARATIVE EXAMPLE B

When the procedure of Example II was repeated with the exception that the application of ethylene glycol was omitted, the average brightness loss due to retention of soil by the carpet samples was 44.5%.

EXAMPLE III—BUTYLENE GLYCOL

When the procedure of Example II was repeated with the exception that a liquid butylene glycol (1,4-butane diol) was substituted for the ethylene glycol, the average brightness loss due to retention of soil by the carpet samples was 27.6%.

EXAMPLE IV—BUTYLENE GLYCOL

When the procedure of Example III was repeated with the exception that the draw pin temperature was maintained at about 190° C., the average brightness loss due to retention of soil by the carpet samples was 30%.

EXAMPLE V—DIETHYLENE GLYCOL

Substantially undrawn polyhexamethylene adipamide fibers consisting of six filaments having a total denier of 465 were drawn at a draw ratio of 4.32 over a ¾-inch diameter draw pin maintained at about 150° C. and then taken up at a speed of 112 feet per minute. The fibers were

in contact with the pin for about 0.2 second. Immediately before being drawn, the fibers were substantially uniformly coated with about 4.77%, based on the weight of the fibers, of liquid diethylene glycol

($HOCH_2CH_2OCH_2CH_2OH$)

at room temperature by a syringe meter pump. Circular tubing was knit from the drawn fibers, blank-dyed, soiled and vacuum-cleaned as in Example I. Using the brightness value measurement technique of Example I, it was found that the retention of soil by the tubing samples had resulted in an average brightness loss of 39%.

COMPARATIVE EXAMPLE C

When the procedure of Example V was repeated with the exception that the application of diethylene glycol was omitted, the average brightness loss due to retention of soil by the tubing samples was 58%.

EXAMPLE VI—TRIETHYLENE GLYCOL

When the procedure of Example II was repeated with the exception that liquid triethylene glycol

($HOCH_2CH_2OCH_2CH_2OCH_2CH_2OH$)

was substituted for the ethylene glycol, the average brightness loss due to retention of soil by the carpet samples was 31.4%.

EXAMPLE VII—TRIETHYLENEGLYCOL

When the procedure of Example II was repeated with the exception that the draw pin temperature was maintained at about 190° C. and the amount of triethylene glycol with which the fibers were coated was about 5%, based on the weight of the fibers, the average brightness loss due to retention of soil by the carpet samples was 33.1%.

COMPARATIVE EXAMPLE D

When the procedure of Example VII was repeated with the exception that the application of triethylene glycol was omitted, the average brightness loss due to retention of soil by the carpet samples was 40.5%.

EXAMPLE VIII—TETRAETHYLENE GLYCOL

When the procedure of Example I was repeated with the exception that the draw pin was immersed in a bath of liquid tetraethylene glycol

($HOCH_2CH_2OCH_2CH_2OCH_2CH_2OCH_2CH_2OH$)

maintained at about 205° C. and the draw ratio was about 4.32, the average brightness loss due to retention of soil by the tubing samples was 37%.

COMPARATIVE EXAMPLE E

When the procedure of Example VIII was repeated with the exception that the application of tetraethylene glycol was omitted, the average brightness loss due to retention of soil by the tubing samples was 53%.

EXAMPLE IX—400 MW POLYETHYLENE GLYCOL

When the procedure of Example II was repated with the exception that liquid polyethylene glycol having a molecular weight of about 400 was substituted for the ethylene glycol and the draw pin temperature was maintained at about 190° C., the average brightness loss due to retention of soil by the carpet samples was 37.5%.

EXAMPLE X—GLYCEROL

When the procedure of Example II was repeated with the exception that liquid glycerol was substituted for the ethylene glycol, the average brightness loss due to retention of soil by the carpet samples was 31.4%.

The degree of lowering of the soil retentiveness of polyamide fibers by the process of this invention may be more fully appreciated by reference to the following table in

which the brightness losses of the foregoing examples are arranged in groups according to draw pin temperatures so that the differences between the results of the examples in such groups are attributable only to the liquid substances with which the fibers were coated in Examples I–X.

Example number	Liquid substance	Draw pin temperature, ° C.	Brightness loss, percent
I	Ethylene glycol (bath)	135	47, 6
A	None	135	60, 4
II	10% ethylene glycol	150	36, 6
III	10% butylene glycol	150	27, 6
VI	10% triethylene glycol	150	31, 4
X	10% glycerol	150	31, 4
B	None	150	44, 5
V	4.77% diethylene glycol	150	39
C	None	150	58
IV	10% butylene glycol	190	30
VII	5% triethylene glycol	190	33, 1
IX	10% 400 M.W. polyethylene glycol	190	37, 5
D	None	190	40, 5
VIII	Tetraethylene glycol (bath)	205	37
E	None	205	53

COMPARATIVE EXAMPLE F

When the procedures of Examples I through X are repeated with the exception that the liquid substances (i.e., the glycerol, alkylene glycols and polyalkylene glycol) are not applied to the fibers until immediately after the drawing of the fibers has been completed, the average brightness losses due to retention of soil by the samples are substantially the same as when the procedures of those examples are carried out without any application of such liquid substances. Accordingly, and although there are no known structural characteristics by which the fibers produced by the process of this invention can be distinguished from the fibers produced by a process which differs from that of this invention only in that such liquid substances are not applied to the fibers until after the drawing of the fibers has been completed, it is apparent that the fibers produced by the present process are characterized by substantially lower soil retentiveness than the fibers produced by a process which differs only in that respect from the process of this invention.

I claim:

1. A process which comprises coating substantially undrawn linear polyamide fibers with at least about 2.5 percent, based on the weight of the fibers, of a liquid substance selected from the group consisting of glycerol, at least one C_2–C_6 alkylene glycol, at least one poly(C_2–C_4 alkylene glycol) having a molecular weight between 106 and 282 and mixtures thereof and then drawing the coated fibers at a draw ratio between about 2.5 and about 6 over a drawing surface heated to a temperature between about 130° and about 220° C.; wherein the time of contact between the fibers and the drawing surface is between about 0.01 and about 0.25 second; said temperature is between about 130° and about 180° C. when the liquid substance is glycerol; said temperature, when the liquid substance is an alkylene glycol, is between about $(120+5n)$° and about $(145+10n)$° C. wherein n represents the number of carbon atoms in said alkylene glycol; and said temperature, when the liquid substance is poly(alkylene glycol), is between about (114.3+0.15 MW)° and about (163.5+0.25 MW)° C. wherein MW represents the molecular weight of said poly(alkylene glycol).

2. A process as defined in claim 1, in which the fibers are coated with from about 2.5 to about 20 percent, based on the weight of the fibers, of said liquid substance.

3. A process as defined in claim 1, in which the polyamide is polyhexamethylene adipamide.

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