

1

2

3,484,186
**COMPOSITION AND METHOD FOR TREATING
TEXTILE PRODUCTS OF POLYAMIDES**
Takeshi Ogawa, Kujochō, and Hiroshi Suzusho, Kyoto,
Japan, assignors to Kanegafuchi Boseki Kabushiki
Kaisha, Tokyo, Japan
No Drawing. Filed Sept. 17, 1965, Ser. No. 488,237
Claims priority, application Japan, Sept. 21, 1964,
39/54,225
Int. Cl. D06m 3/30, 3/42
U.S. Cl. 8—130.1 7 Claims

ABSTRACT OF THE DISCLOSURE

An agent for treating polyamide textile materials comprising an addition compound of a lactam or oligomers thereof and polyalkylene glycol and a swelling agent such as a phenol for the polyamide. The present disclosure also provides for a method of treating a polyamide textile material with the aforementioned agent by applying an aqueous solution of the agent to the polyamide and then heat treating the polyamide at a temperature of about 110° C. to 160° C.

This invention relates to an agent for improving the properties of textile materials of polyamides and also to the treatment of polyamide textile materials. More particularly this invention relates to an agent and method for improving the properties, particularly hygroscopic, antistatic and other properties of textile materials of polyamides.

The term "polyamides" used throughout the specification and claims is intended to mean synthetic linear polyamides or polymeric carboxylic acid amides such as may be prepared by polymerizing amino carboxylic acids or their amide forming derivatives or by polymerizing diamines with dicarboxylic acids, typical being conventional "nylon" polymers.

The term "polyamide textile materials" or "textile materials of polyamides" or the like as used in the specification and claims is intended to include filaments, yarns, knit-fabrics, woven fabrics and made-up articles of clothing, consisting of or containing fibers of polyamides.

It is well known that polyamides have excellent properties for use as textile materials. However, conventional polyamide textile materials have some drawbacks, among which most noticeable is poor hygroscopicity. It is well known that polyamide textile materials are less hygroscopic than similar products of natural fibers and, therefore, are not suitable for use in lingerie, underwear and the like, because they prevent the dissipation of sweat which makes the wearer uncomfortable and susceptible to skin diseases. Further, polyamides are so readily electrically charged that articles of clothing made thereof are readily soiled and in addition render the wearer uncomfortable.

Various attempts have heretofore been made to solve the above problems, and various hygroscopic treating agents and antistatic agents are commercially available. However, most of these agents are not satisfactory because of their relatively poor durability or fastness, particularly against laundering. It is known to impart laundering-resistant hygroscopic properties to polyamide textile materials by the methylation, alkoxymethylation, hydroxyalkylation, etc. of the polyamides, but these methods have certain disadvantages. Thus the hand of the finished product is rendered rough, or the strength of the product is reduced.

Therefore, it is an object of this invention to provide an agent and method for rendering polyamide textile materials hygroscopic and antistatic without those accompanying drawbacks encountered in conventional treating agents and methods.

It is another object of this invention to provide polyamide textile materials having improved hygroscopic and antistatic properties which are durable and resistant to laundering and having desirable properties such as hand, strength, etc. without being adversely affected.

Other objects, advantages and features of this invention will be apparent from the following detailed description.

Briefly, the agent for treating polyamide textile materials according to this invention comprises at least one compound selected from the group consisting of addition compounds of lactams or their derivatives and polyalkylene glycols and at least one compound selected from swelling agents for polyamides. In using the agent, it is diluted with water and the solution is applied to polyamide textile material, which is then heat treated at an elevated temperature.

Also, briefly, the method for treating polyamide textile materials comprises treating the textile materials with an aqueous solution of at least one compound selected from the group consisting of addition compounds of lactams or their derivatives and polyalkylene glycols, in the presence of a swelling agent for polyamides, and then heat treating the textile materials at an elevated temperature.

In carrying out the invention those lactams which are used in the production of polyamides are employed, and ϵ -caprolactam is preferred. By derivatives of lactams, we mean oligomers of lactams. However, if the degree of polymerization is too high in the oligomers the adsorptive affinity for polyamides would be low and the hand or feel of the treated product would be adversely affected. Therefore, in case of caprolactam oligomers those not higher than pentamer are preferable.

As for polyalkylene glycols, those having 2-4 (more preferably 2-3) carbon atoms in the alkylene oxide unit are preferred. Thus, ethylene glycol and propylene glycol are most preferable in this invention. If the number of carbon atoms is higher than 5, the hydrophilic property of the resulting addition compound is lowered and consequently no noticeable improvement in the hygroscopicity would be obtained in the textile product treated according to the present invention.

According to the invention an addition compound of a lactam or its oligomer and polyalkylene glycol is employed. In other words, a lactam or its oligomer is added with alkylene oxide such as ethylene oxide or propylene oxide. If the number of moles of alkylene oxide is too high the resulting addition compound would be so highly soluble in water that the durability, particularly resistance to laundering of the effect of treatment would be lowered. It has been found to be preferable that a lactam or its derivative is added with about 2-10 moles of alkylene oxide.

The addition compounds may be prepared in a conventional manner. Thus, for example, alkylene oxide is introduced into caprolactam in a pressure vessel under pressure and heating. As a more particular example, a predetermined amount of ϵ -caprolactam is charged in a pressure vessel and the space within the vessel is replaced with nitrogen gas. While heating (120-160° C.) and under pressure (1-1.5 atm.) a desired amount of ethylene oxide is introduced. The reaction is completed in about 3-4 hours and an addition compound of ϵ -caprolactam and alkylene oxide is formed.

Swelling agents to be used in this invention are those for polyamides. Among the useful swelling agents are phenol, benzyl alcohol, cyclohexanol, phenolsulfonic acid, formic acid, acetic acid and monochloroacetic acid.

The relative amount of the addition compound and swelling agent is not so critical, but it is generally preferable to use 0.1-1 part by weight of the swelling agent per part of the addition compound.

In treating polyamide textile materials according to this invention, the treating agent is diluted with water. The concentration is not critical and may vary over a wide range depending upon the particular method of application of the solution to the textile materials and also depending upon the time of immersion and pick up. However, in general, the agent is diluted with water so that about 0.05 to 5 g. of the addition compound will be contained in 1000 ml. of the treating solution.

The treating solution may be applied to the textile materials in any suitable manner such as padding, dipping and spraying. The time of immersion and degree of squeezing (i.e. pick up) may be so adjusted, depending upon the concentration of the treating solution, that at least 0.008% by weight (based on the textile material to be treated) of the addition compound is adsorbed on the textile material.

The textile material impregnated with the treating solution in the above mentioned manner is then subjected to a heat treatment. If desired, the textile material may be predried prior to the heat treatment. The predrying may be conducted at a temperature not higher than 100° C. The heat treatment is conducted at a temperature higher than 100° C., preferably at a temperature of from 110° C. to 160° C. The heat treatment may be carried out in a wet state or dry state. The time for the heat treatment may vary over a wide range from several seconds to several minutes depending upon the temperature of the heat treatment. It should be understood that the shorter the treating time, the higher the temperature should be.

After the heat treatment, the textile material is washed to remove the swelling agent, and then dried.

It is well known that polyamide textile materials are low in hygroscopicity due to the fact that polyamides have only a small number of active functional groups. In fact, the standard moisture of typical "nylon" fibers is only about 4%. In contrast thereto, the polyamide textile materials (composed only of polyamide) treated according to this invention show the moisture content of about 8% which is comparable with the standard moisture content of cotton fiber, and the antistaticity is improved correspondingly. Furthermore, the polyamide textile materials treated according to this invention not only retain the initial strength but have hand or feel which is comfortable when used as underwear and other garments. The polyamide textile materials treated according to this invention are free from electro-discharging due to friction. More importantly, these desirable properties remain valid for a prolonged period of time and are not lost even after repeated laundering.

Although the mechanism by which the excellent and durable hygroscopic and antistatic effect is obtained in the treatment of this invention is not fully ascertained, it is believed that the following phenomena would take place.

As mentioned before, the presence of a swelling agent is essential in the present invention. The swelling of polyamide starts in the amorphous region of the polyamide and proceeds into the mimetic crystal region. In addition, the swelling also proceeds into the intermicelle space with the progress of time. It is deemed that a melting and transition phenomenon would take place during the above mentioned swelling and simultaneous heat treatment. Stated differently, unstable micro-crystallite would be molten at the heat treating temperature and recrystallized into more stable crystals. Then there would occur the cohesion and orientation of the addition compound on the recrystallized surface and in the intermicelle space enlarged due to the above mentioned swelling, so that there would be formed auxiliary valency force bonds such as hydrogen bond. In this way, the low density region of the polyamide is filled or clutched, resulting in hygroscopic and antistatic properties which are highly resistant to laundering.

Since the addition compound of a lactam or its deriva-

tive and an alkylene glycol has a surface active action, the dispersion and penetration of the swelling agent into the polyamide fiber are facilitated. The rapid penetration of the swelling (and hence rapid swelling of polyamide fiber) is conducive to the correspondingly rapid penetration of the addition compound. Therefore, there is a synergistic effect which would ensure prompt and effective reaction not seen in conventional methods.

The invention will be more concretely described by referring to the following examples which are given for illustration only and not for limitation of the invention in any way.

In these examples, the wash test was conducted by washing a sample fabric (1.36 kg.) with 68 l. of water containing 0.1% of non-ionic detergent at 40° C. for 15 minutes in a jet-type washing machine. Then the fabric was washed twice with hot water of 95° C. for 10 minutes and dehydrated by a centrifuge and dried.

The hygroscopicity was determined by water diffusion time which was measured by drawing a circle (3 cm. in diameter) on the sample fabric and dropping 0.02 cc. of 2% aqueous solution of potassium permanganate upon the centre of the circle through the tip of a burette placed at a distance of 5 cm. above the fabric. The time (seconds) required for the red-purple color of the solution to reach the circumference of the circle was measured. The measurement was repeated 5 times and the mean average value was taken.

EXAMPLE 1

A "nylon" knit fabric was immersed in an aqueous solution (25° C.) containing 0.1 g./l. of an addition compound of 1 mol caprolactam and 10 mols of ethylene oxide and 0.05 g./l. of benzyl alcohol, and was squeezed with a mangle to a pick up of about 80% by weight (based on the weight of the fabric). After pre-drying at 100° C. for 3 minutes, the fabric was dry-heat treated at a temperature of 135° C. \pm 5° C. for 5 minutes and was then subjected to soaping to remove benzyl alcohol. The hygroscopicity of the treated fabric was 1.1 seconds, and 6.7 seconds even after 5 washings, whereas the hygroscopicity of the non-treated fabric was 30.5 seconds.

EXAMPLE 2

A "nylon" knit fabric was immersed in an aqueous solution (20° C.) containing 0.38 g./l. of an addition compound of 60 parts by weight of a mixture of dimer and trimer of caprolactam and 31 parts by weight of ethylene oxide and 0.3 g./l. of phenol, and was squeezed, pre-dried and heat treated in the same manner as in Example 1. The fabric was subjected to soaping to remove the phenol and dried. The fabric so treated had a hygroscopicity of 2.5 seconds while the hygroscopicity of the non-treated fabric was 100 seconds or more.

EXAMPLE 3

An aqueous solution containing 0.8 g./l. of an addition compound of a mixture (30 parts by weight) of dimer and trimer of caprolactam and propylene oxide (30 parts by weight) and 0.1 g./l. benzyl alcohol was applied to 70-denier "nylon" filaments by means of rotating roller so that the solution is retained on the filaments in an amount of about 80% by weight based upon the filaments. The filaments were false-twisted on a false-twister and at the same time were heat set at a temperature of 150° C. \pm 2° C. Then the filaments were subjected to soaping to remove benzyl alcohol. There were obtained crimped yarns having an excellent hygroscopicity.

By using the treated yarns, a "nylon" taffeta having a density of 102 warps per inch and 73 wefts per inch was made. The hygroscopicity of this fabric was 1.5 seconds, while a similar fabric woven with non-treated yarns was 90 seconds or more in hygroscopicity.

EXAMPLE 4

A mixed fabric composed of "nylon" yarn (warps) and polyester yarn (weft) was immersed in a mixture of 0.2 g./l. of an addition compound of ϵ -caprolactam 1 mol and ethylene oxide 5 mols and 0.1 g./l. of cyclohexanol, and then treated in the same manner as in Example 1. The hygroscopicity of the treated fabric was 3.5 seconds, while that of non-treated fabric was 90 seconds or more.

What we claim is:

1. A composition for treating polyamide textile materials to render such materials more antistatic and hygroscopic, which consists essentially of (1) an addition compound of a lactam or an oligomer thereof selected from the group consisting of a dimer, trimer, tetramer and pentamer of caprolactam and an alkylene oxide having 2-4 carbon atoms in the alkylene oxide unit, the molar ratio of the lactam to the alkylene oxide being from 1:2 to 1:10, and (2) a swelling agent for the polyamide, wherein the swelling agent is present in an amount of 0.1-1 part by weight per part of the addition compound.

2. A composition as claimed in claim 1 wherein the swelling agent is selected from phenol, benzyl alcohol, cyclohexanol, phenolsulfonic acid, formic acid, acetic acid and monochloroacetic acid.

3. A method for treating polyamide textile materials to increase the antistatic and hygroscopic properties thereof, which comprises applying to the textile material an aqueous solution consisting essentially of (1) an addition compound of a lactam or oligomers thereof selected from the group consisting of a dimer, trimer, tetramer and a pentamer of caprolactam and 2-10 mols of a poly-alkylene glycol and (2) a swelling agent for the poly-

amide in an amount of 0.1-1 part by weight, per part of the addition compound, heating the textile material at a temperature higher than 100° C. and then washing the textile material.

4. A method as claimed in claim 3 wherein the heat treatment is conducted at a temperature of 110° C. to 160° C.

5. A method as claimed in claim 3 wherein the alkylene glycol is selected from those having 2-4 carbon atoms in the alkylene oxide unit.

6. A method as claimed in claim 3 wherein the swelling agent is selected from phenol, benzyl alcohol, cyclohexanol, phenolsulfonic acid, formic acid, acetic acid and monochloroacetic acid.

7. A method as claimed in claim 3 wherein the aqueous solution is applied to the textile material so that at least 0.008% by weight of the addition compound is adsorbed on the textile material.

References Cited

UNITED STATES PATENTS

3,053,609	9/1962	Miller	8-130.1 X
3,269,965	8/1966	Johnson	260-78 X
3,312,753	10/1967	Bailey	260-860 X

WILLIAM D. MARTIN, Primary Examiner

J. E. MILLER, JR., Assistant Examiner

U.S. Cl. X.R.

117-138.8, 139.5; 252-8.9