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2,855,267

PROCESS OF IMPROVING THE ACID DYE RECEPTIVITY OF NYLON TEXTILES BY REACTION WITH SPECIFIC ORGANIC AMINES AND PRODUCTS PRODUCED THEREBY

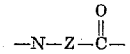
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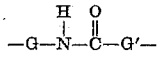
7 Claims. (Cl. 8—115.5)

This invention relates to a textile produced from a polyamide. More particularly it is concerned with a process for treating such a textile to improve its dyeability with an acid dye.

By the term "textile produced from a polyamide" is meant a fabric, fiber, filament, yarn, pellicle, flock and the like produced from a linear polyamide containing recurring units of the formula:



wherein Z is a member of the class consisting of a divalent hydrocarbon radical and a divalent radical of the formula



wherein G and G' are divalent hydrocarbon radicals. Typical polyamides and processes for their production are described in United States Patents Nos. 2,071,250, 2,071,253 and 2,130,948.

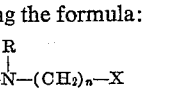
While it is known that textiles produced from a polyamide have some affinity for acid dyes, difficulty is often experienced in dyeing such textiles to deep shades. The problem is particularly acute when an attempt is made to dye such a textile with acid dye when the polyamide from which it is prepared has been polymerized in the presence of an acid-type viscosity stabilizer, as for instance, an excess of dibasic acid, as taught in U. S. Patent No. 2,174,527.

It is an object of the present invention to prepare a textile produced from a linear polyamide which will dye to deep shades with acid dye.

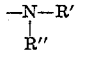
Another object is to provide a process for the treatment of a textile produced from a polyamide to increase its susceptibility to acid dye.

These and other objects will become apparent in the course of the following specification and claims.

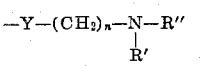
In accordance with the present invention a textile produced from a polyamide is heated in contact with an organic compound having the formula:



wherein X is a radical of the class consisting of:



and



wherein Y is tertiary amino from the class consisting of alkyl amino, aryl amino and piperazino, R, R' and R'' being members of the class consisting of hydrogen and hydrocarbon radicals and n is an integer of at least 2.

The following examples are cited to illustrate the invention. They are not intended to limit the invention in any way. In the examples cited, dye bath ingredient composition is reported in parts by weight based on the weight of the textile dyed.

Example I

A five gram sample of a jersey stitch knit tubing made from 70 denier, 34 filament polyhexamethylene adipamide yarn having a ½ Z twist to the inch and produced in accordance with the teachings of Heckert in U. S. Patent 2,273,104 and containing 0.3% TiO_2 as a delusterant is immersed in a 20% aqueous solution of hexamethylene diamine at room temperature. After removal of excess solution by wringing, the tubing is heated in a 218° C. nitrogen atmosphere for one hour. Thereafter it is immersed in a dye bath for 60 minutes at 95° C. (bath to fabric ratio of 50:1) the dye bath having the composition:

	Parts
Anthraquinone Green GN (C. I. #1078)	0.15
Water	250.
Acetic acid	0.1
Sodium alkyl sulfate	0.1

Excess dye solution is squeezed from the sample and it is dried at room temperature. The sample is observed to be evenly dyed a deep shade of green.

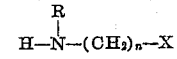
A control sample, without treatment with hexamethylene diamine as taught herein, is observed to accept only one third as much dye when dyed in the bath and under the dyeing conditions described above.

Example II

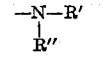
A four inch length sample of the knit tubing of Example I is immersed in a 20% aqueous solution of N,N'-bis (aminopentyl) piperazine for one minute at room temperature. After removal of excess solution by wringing, the sample is heated in a 218° C. nitrogen atmosphere for 30 minutes. Thereafter it is dyed with the bath and according to the technique of Example I. The sample is dyed to an intense shade of green. A control sample, not pretreated with the piperazine derivative, accepts only a pale green coloration when dyed under the same conditions.

While applicant does not wish to be bound by any theory of operation, it is postulated that the polyamide modification in accordance with the present invention involves interchange between active hydrogen bearing nitrogen of the additive and the amide groups of the polyamide fiber.

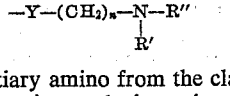
As previously mentioned the modifying compound may be any compound having the formula:



wherein X is a radical of the class consisting of:



and



wherein Y is a tertiary amino from the class consisting of alkyl amino, aryl amino and piperazino, R, R', and R'' being members of the class consisting of hydrogen and hydrocarbon radicals and n is an integer of at least 2.

As will be obvious from the above, the diamine contains at least one active hydrogen attached to nitrogen. Specific embodiments include ethylenediamine, N-alkyl ethylenediamine, N,N-dialkyl ethylenediamine, N,N'-dialkyl ethylenediamine, N,N,N'-trialkyl ethylenediamine, the corresponding aryl substituted materials and the homologously related polyalkylene diamines and deriva-

tives thereof such as tetramethylene diamine, pentamethylene diamine, hexamethylene diamine and the like. The chain of the diamine may be interrupted by substituted aza linkages. Such materials include bis(aminoalkyl) alkylamine, bis(aminoalkyl) arylamine, bis (N-alkylaminoalkyl) alkylamine and the like. As exemplified the nitrogen within the chain of the diamine may be supplied by piperazine. Those compounds are preferred wherein n is no greater than about 10 and in which the nitrogen substituents are hydrogen or lower alkyl.

Where the modifying substance is water-soluble, it may be applied to the textile from an aqueous solution and this will usually be possible. However, the nature of the solvent is not critical. Non-phenolic organic solvents are suitable. Among those which may be used are ethanol, acetone, and methyl ethyl ketone. Thus while it is convenient to apply the modifying material from solution, this is not necessary where the modifying compound is a liquid. In such a case it may be applied directly, without dilution, to the textile which is treated. The modifying liquid may be contacted with the textile by any conventional means such as by spreading or dipping the textile in a vat of the modifying compound such as in a beck or jig. The amount of modifying compound applied to the textile will affect its acid dye receptivity. To attain uniform modification, it is convenient to completely wet the textile with a modifying solution and thereafter remove excess liquid by wringing, centrifugal action, or the like.

When diprimary amines, disecondary amines or primary-secondary diamines are used as modifying amines, their concentration is adjusted so that the sum of unreacted primary and/or secondary amine end-groups applied to the treated yarn is from 40 to 200 equivalents per million grams of yarn. When the modifiers contain tertiary amino groups in the chain, an additional number of tertiary amine dye sites are added.

The textile moistened with the modifying liquid is subjected to an inert atmosphere at a temperature of at least about 150° C. and no higher than the melting point of the fiber. A temperature range of from about 175° C. to about 15° below the melting point of the polyamide is preferred. At these temperature ranges, an exposure period of from about 5 to about 60 minutes is adequate. Longer periods may be employed when a high degree of modification is desired without substantial loss of molecular weight. Somewhat higher temperatures are suitable provided the temperature range does not approach the decomposition point of the textile treated. The inert atmosphere may be supplied by nitrogen or other inert gas. The heating may also be performed in a low-pressure chamber, a pressure of about 10–20 mm. providing adequate protection. Still another method of heating is to pass the treated object through a tank containing a molten metal such as Woods metal or an inert liquid such as a silicone.

While it is most convenient to remove excess modifying liquid from the textile and thereafter heat the moistened textile in an inert gaseous atmosphere, as an alternative procedure, the moistened textile may be heated in an inert solvent or in a solution of the modifier if the modifier solvent is inert to the textile being treated. For example, a hydrocarbon may be used provided that steps are taken to avoid excessive loss of solvent by evaporation and contact of the polyamide with air. Still another method for obtaining the modification is to heat the polyamide textile at the recited temperatures in an oxygen-free atmosphere containing the modifier in the gaseous or dispersed state. With this procedure the extent of modification is controlled by adjusting the pressure of the modifier, temperature, and time of heating.

After the textile has been modified it is ready for application of dye. Among acid dyes which are suitable may be mentioned the sodium bisulphite salt of para-sulfobenzene-azo-beta-naphthol (C. I. 152), the toluene-para-sulphonyl esters (C. I. 430), the sodium salt of toluene-para-sulphonyl-ether of 4-para-hydroxybenezene-azo - 1 - para - chloro - orthosulpho - phenyl - 3 - methyl-5-hydroxy-pyrazol (C. I. 642), the sulphonic acids of dialkylamino - phenylamino - phenyl - naphthophenazonium chloride (C. I. 833) and the sodium salt of 1:4-di-ortho-sulpho-para-tolyl-aminoanthraquinone (C. I. 1078). These dyes are applied by conventional techniques.

As previously mentioned, any linear polyamide containing recurring units of the formula:

wherein Z is a member of the class consisting of a divalent hydrocarbon radical and a divalent radical of the formula



wherein G and G′ are divalent hydrocarbon radicals, may be employed. Such polyamides may be prepared in accordance with procedures known to the art, e. g., by the polymerization of diamines and dibasic acids or by the polycondensation of an amino acid, or the polymerization of lactams. Thus, those polyamides prepared by the polymerization of diamines such as tetramethylene, pentamethylene, hexamethylene, methyl hexamethylene, decamethylene, tridecamethylene, octadecamethylene, and para-xylylene diamine with such dibasic acids as malonic, glutaric, methyl glutaric, adipic, suberic, azelaic, sebacic, brassylic, tetradecanedioic, octadecanedioic, p-phenylene diacetic, terephthalic, and hexahydroterephthalic acid are suggested. Suitable amino acids which may be polymerized to fiber-forming polyamides include 6-amino caproic acid, 8-amino caprylic acid, 17-amino heptadecanoic acid and the like including their lactams.

The physical form of the textile at the time it is modified is not critical. Thus, it may be in the form of a finished fabric. The process may also be applied to polyamide films, staple, flock, continuous filament, spun yarn, mixed yarns, such as a combination of polyamide staple and wool, polyamide staple and cotton, polyamide staple and rayon, and the like.

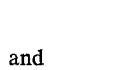
Many other modifications will be apparent to those skilled in the art from a reading of the above disclosure without a departure from the inventive concept.

What is claimed is:

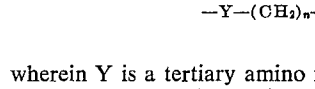
1. A process for increasing the acid dye receptivity of a textile produced from a fiber-forming synthetic linear polycarbonamide having recurring amide groups as integral parts of the main polymer chain, which comprises heating the said textile in an inert medium in contact with an organic compound having the formula:



wherein X is a radical of the class consisting of:



and



wherein Y is a tertiary amino radical from the class consisting of lower alkyl amino, and piperazino, R, R′ and R″ are a member of the class consisting of hydrogen and lower alkyl and n is an integer of from 2 to 10 at a temperature of from about 150° C. to the melting point of the fiber.

2. The process of claim 1 wherein the textile is moistened with the modifying compound and heated in an inert gas.

3. The process of claim 1 wherein the organic compound is hexamethylene diamine.

4. The process of claim 1 wherein the organic compound is N,N'-bis(aminopentyl)piperazine.
5. The textile produced by the process of claim 1.
6. The textile produced by the process of claim 3.
7. The textile produced by the process of claim 4.

References Cited in the file of this patent

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