A = UNTREATED POLYESTER FIBERS
B = POLYESTER FIBERS TREATED ACCORDING TO EXAMPLE 1

Reflection in %

7000 6500 6000 5500 5000 4500 4000

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BY

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PROCESS FOR INCREASING THE COLORABILITY OF POLYESTER TEREPHTHALATE FIBERS AND THE PRODUCTS OBTAINED THEREBY

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Filed Sept. 3, 1957, Ser. No. 681,880

Claims priority, application Germany Feb. 20, 1957

12 Claims. (Cl. 8—115.5)

This invention relates to polyester fibers and is more particularly directed to a process for improving and increasing the colorability of polyester fibers.

The term “fibers” is used in this specification and the appended claims in its broadest sense and is deemed to include filaments, threads, yarns and bristles either wholly composed of or comprising polyester, fabrics made therefrom and articles made from such fabrics.

Owing to their but slight swelling capacity and activity, polyesters—as is known—take dispersion dyes and dyestuffs only slightly and thus are colored or dyed by such dyes to a negligible extent only. For this reason the dyeing of polyester fibers with dispersion dyes cannot be effected by ordinary dyeing processes.

It is known that dyes of the two classes may be obtained on polyester fibers, if the dyeing baths are admixed with substances that exert a swelling action on the fibers. Such substances are generally referred to as “carriers.”

Further, it is known that certain polyester fibers may be obtained by performing the dyeing procedure at elevated temperatures of above 100° C. in specially constructed dyeing apparatus.

Both dyeing procedures referred to possess, however, considerable disadvantages as follows: The so called “carrier” process referred to constitutes decidedly a last, and very unsatisfactory so, resort, since the addition of the carriers to the one hand renders the dyeing procedure more expensive and on the other hand the dyeing bath or liquor containing the swelling agents usually has to be discarded as waste already after a single utilization. Further, colorings obtained by this method are commonly not as fast to light as required or desired and fade easily.

The second procedure referred to, i.e., dyeing at elevated temperatures above 100° C., obviously inherently requires technical expenditure and apparatus which ordinarily is not available to smaller dye works.

In view of the above remarks reflecting the state of the art, it appears obvious that it would be most desirable to impart to polyester fibers already at the time of their formation the facility readily to take dispersion dyes, thus obviating cumbersome subsequent auxiliary measures and processes.

Accordingly, it is an object of this invention to provide a process by means of which the colorability or capacity to take dyes of polyester fibers is considerably improved.

A further object of the invention is generally to improve processes relating to the dyeing of polyester fibers.

In accordance with the present invention it has been found that the capacity of polyester fibers to take dyes is considerably increased if the polyester fibers are treated with N-cyclohexylidene-hydroxyethylamine, N-cyclohexylidene-dihydroxyethylamine, N-methylcyclohexylidene-hydroxyethylamine or N-methylcyclohexylidene-dihydroxyethylamine. The cyclohexylidene derivatives are soluble in water and consequently may be employed in the form of aqueous solutions. The methylycyclohexylidene derivatives, on the other hand, are not soluble in water and accordingly they are first dissolved in alcohol or other suitable organic solvents having a boiling point of above 80° C. Whereafter these solutions are made to act on the polyester fibers.

The compounds enumerated above and employed in accordance with this invention for the purpose of increasing the colorability of polyester fibers, may be obtained in a simple manner by reacting cyclohexanone or methylycyclohexanone with monoethanolamine or diethanolamine, respectively.

With a view to obtaining the desired compounds in pure form it may be required in some instances to effect distillation. However, for the purposes of the inventive process, it is not absolutely necessary to use the compounds in pure form.

According to a further feature of this invention, it has been ascertained that almost the same beneficial, colorability improving effect on polyester fibers is obtained, if mixtures of technical triethanolamine with cyclohexanone or methylycyclohexanone, respectively are employed. The manner in which these compounds act upon the polyester fibers is at the present time unknown. It is certain, however, that no swelling procedure is involved and that the respective compounds are not deposited on the surface of the fibers, because the increased capacity of the polyester fibers to take dyes, if treated in accordance with this invention, is permanent and remains entirely even after thorough washing of the fibers and also after prolonged storage.

The treatment procedure of the polyester fibers with the compounds may be effected in different ways and under varying conditions. In accordance with one embodiment of the inventive process the fibers, in form of endless threads, tufts or yarn are treated with the suitable cyclohexanone derivatives in aqueous solution of 5—20% concentration. Another possibility is to dissolve the methylycyclohexanone derivatives—or the cyclohexanone compounds—in organic solvents so as to obtain solutions of 1—20% concentration, whereafter the fibers are subjected to the action of the solutions. It is furthermore possible to treat the fibers with the undiluted compounds, i.e., with the compounds in liquid form or substantially 100%. The last mentioned procedure has the advantage that the treatment time of course will be shortened considerably.

In all three procedures the treatment should be effected at a temperature of about 80—100° C. The treatment time depends on the respective concentration of the compounds.

With a view to demonstrating the increased colorability of polyester fibers that have been subjected to the inventive process as distinguished from untreated polyester fibers, reference is had to the accompanying graphic representation, forming part of this application. This graphic representation was obtained in the following manner: A batch of treated and untreated polyester fibers, respectively, was dyed with 3% of the red dye “Cillionechrot” at a bath ratio of 1:50. The dyeing was carried out at boiling temperature for 1 hour. The dyed fibers were then rinsed, whereafter the fibers were...
further treated for about 30 minutes at boiling temperature with 2 grams per liter of a non-ionic detergent at a bath ratio of 1:50. After rinsing and drying, the reflection of the thus treated fiber material was measured against a standard white at different wave lengths of visible light. The measurements were effected by means of a Zeiss photometer with Ullbricht's ball. The smaller the reflection, the more intensive is the coloration. As will be readily gathered from the graph, the reflection of the treated fibers is much more insignificant than that of the untreated ones and, consequently, the coloration of the former much more pronounced.

The invention will now be described by several examples, it being understood, however, that these examples are given by way of illustration rather than by way of limitation and that many variations and changes are possible without departing in any way from the spirit and scope of this invention as recited in the appended claims.

Example 1

10 grams of polyester in tuft form are heated for about 30 minutes and at about 90° C. in 250 cubic centimeters of an aqueous solution of N-cyclohexyldiene-hydroxyethyamine of 20% concentration. The tuft is then well rinsed with water. The rinse water is removed by squeezing or centrifuging, whereafter the tuft is dried in a drying oven at about 60–70° C.

The tuft thus obtained exhibits a greatly increased colorability, which corresponds to the accompanying graphic representation previously referred to.

Example 2

10 grams of polyester in tuft form are immersed in a N-methylcyclohexyldiene-hydroxyethyamine bath at 15 minutes at 90–95° C. The tuft is then washed with a suitable washing-active substance (detergent) and well rinsed. For the purpose of removing the rinse water the tuft is squeezed or subjected to centrifuging, whereafter it is dried in a drying oven at 60–70° C.

The improved colorability of the polyester fiber tuft thus obtained corresponds to the accompanying graphic representation.

Example 3

10 grams of polyester in tuft form are heated for 2 hours in a vessel fitted with reflux cooler and containing 250 cubic centimeters of a 5% solution of N-methylcyclohexyldiene-hydroxyethyamine in ethylene chloride. The tuft is subsequently removed from the vessel, washed with a suitable detergent and rinsed. The tuft is then squeezed or centrifuged and dried in a drying oven at about 60–70° C.

The improved colorability of the polyester fiber tuft thus obtained corresponds to the accompanying graph.

Example 4

10 grams of polyester in tuft form are heated for about 30 minutes at about 90° C. in 250 cubic centimeters of a 20% aqueous solution of N-cyclohexyldiene-di-hydroxyethyamine. The tuft is then well rinsed with water, squeezed and dried in a drying oven at 60–70° C.

The tuft thus obtained possesses an improved colorability which corresponds to that of the tufts obtained according to Examples 1–3.

Example 5

10 grams of polyester tuft are heated to boiling for about 2 hours in a vessel fitted with reflux condenser and containing a 5% solution of N-methylcyclohexyldiene-di-hydroxyethyamine in ethylene chloride. The thus treated tuft is then washed in a suitable detergent and well rinsed. After squeezing of the tuft, it is dried at 60–70° C.

The tuft thus obtained has a markedly improved colorability which corresponds to that of the tufts obtained according to Examples 1–4.

Example 6

10 grams of polyester fibers in tuft form are heated for about 30 minutes at about 90° C. in 250 cubic centimeters of a 20% aqueous solution of the mixing product of 1 mole of triethanolamine and 1 mole of cyclohexanon. The thus treated tuft is then rinsed with water, squeezed, and dried in a drying oven at 60–70° C. The tuft thus obtained possesses substantially the increased colorability of the tufts obtained according to Examples 1–5.

Example 7

10 grams of polyester fibers in tuft form are heated to boiling for about 2 hours in a vessel fitted with reflux condenser and containing 250 cubic centimeters of a 5% solution of the product of 1 mole of triethanolamine and 1 mole of methylcyclohexan in ethylene chloride. After separation of the solution, the tuft is washed in a suitable detergent and well rinsed. After squeezing, the tuft is dried at 60–70° C.

The tuft thus obtained possesses substantially the improved colorability of the tufts obtained by the preceding examples.

The term "polyester," as used in this specification and the appended claims, is deemed to mean high polymer products which are obtained by dicarboxylic acids, preferably terephthalic acid, reacting with glycols, preferably ethylene glycol. These esters are able to form fibers, which are available in the trade for example under the designations "Terylene" (trademark) and "Dacron" (trademark).

The polyester tuft used in Examples 1 to 7 has the following chemical composition:

\[
\text{CH}_\text{CH}_\text{BOO}\cdots \text{COO} \cdot \text{CHCH}_\text{CH}_\text{COO}\cdots \text{COO} \cdot \text{CHCH}_\text{CH}_\text{COO}\cdots \text{COO}
\]

wherein \(n\) stands for a number which results in a molecular weight of the composition between 8000 to 10,000.

What is claimed is:

1. The process for improving the colorability of fibers, which comprises contacting polyethylene terephthalate fibers at a temperature of about between 80–100° C. with a treating agent consisting essentially of a N-methylcyclohexyldiene-di-hydroxyethyamine dissolved in an organic solvent having a boiling point above 80° C.

2. In a process as claimed in claim 1, wherein said solvent is ethylene chloride.

3. In a process as claimed in claim 1, wherein the concentration of said compound in said solvent is between about 5–20%.

4. The process for increasing the colorability of polyethylene terephthalate fibers which comprises treating said fibers at a temperature of 80°–100° C. with a treating agent consisting essentially of a solvent and N-cyclohexyldiene-hydroxyethyamine.

5. Polyethylene terephthalate fibers obtained according to the process of claim 4.

6. The process for increasing the colorability of polyethylene terephthalate fibers which comprises treating said fibers at a temperature of 80°–100° C. with a treating agent consisting essentially of a solvent and N-cyclohexyldiene-di-hydroxyethyamine.

7. Polyethylene terephthalate fibers obtained according to the process of claim 6.

8. The process for increasing the colorability of polyethylene terephthalate fibers which comprises treating said fibers at a temperature of 80°–100° C. with a treating agent consisting essentially of a solvent and N-methylcyclohexyldiene-hydroxyethyamine.

9. Polyethylene terephthalate fibers obtained according to the process of claim 8.

10. The process for increasing the colorability of poly-
ethylene terephthalate fibers which comprises treating said fibers at a temperature of 80°-100° C. with a treating agent consisting essentially of a solvent and N-methylcyclohexylidene-di-hydroxyethylamine.

11. Polyethylene terephthalate fibers obtained according to the process of claim 10.

12. The process for improving the colorability of polyethylene terephthalate fibers, which comprises contacting polyethylene terephthalate fiber at a temperature of about between 80–100° C. with a treating agent consisting essentially of N-methylcyclohexylidene-hydroxy ethylamine dissolved in an organic solvent having a boiling point above 80° C.

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CERTIFICATE OF CORRECTION

Patent No. 3,022,131

February 20, 1962

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It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 4, line 43, before "fibers" insert -- polyethylene terephthalate --.

Signed and sealed this 19th day of June 1962.

(SEAL)

Attest:

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