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2,934,397

PROCESS FOR DYEING POLYESTER FIBER

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No Drawing. Application October 26, 1955
Serial No. 543,038

8 Claims. (Cl. 8--55)

This invention relates to an improved process for dyeing polyester fibers such as polyethylene-terephthalate or cellulose triacetate.

The hydrophobic nature of fibers of the above type ordinarily makes their dyeing a difficult practical problem. Thus, while it has been observed that disperse dyes which were originally developed for cellulose acetate have some affinity for polyester fibers, the latter are much more resistant to dyeing with such dyes than is cellulose acetate. Under normal dyeing conditions almost no disperse color is absorbed by polyester fiber. At the boil, a few acetate dyes color the fabric somewhat, but penetration is very poor. There are two methods known to increase the penetration of the disperse color into the polyester fiber: (a) pressure dyeing at elevated temperatures and (b) use of a carrier at or near the boil. A carrier is a chemical assistant having limited water solubility, but which aids in absorption and penetration of the dye into the fabric.

Several carriers are available which aid in dyeing polyester fibers with disperse colors. Benzoic acid and salicylic acid for example have been used. Ortho-phenylphenol is another carrier which is customarily used where heavy shades of disperse dyes on polyester fibers are desired. However, use of the currently available carriers presents several problems since they are all deficient in many desirable characteristics, for instance from the standpoint of cost, toxicity, odor, ease of removal from the fabric, ability to promote transfer of colors (dye levels) or availability. Conventional carriers used heretofore are also generally injurious to the light fastness of the disperse dyes used and, because most of them are liquid under dyeing conditions, they tend to cause spotting of the fabric and blotchiness.

It is accordingly an object of this invention to provide a process for dyeing polyester fibers with disperse dyes in which level, heavy shades of dye, having good light fastness, can be obtained on the fabric without spotting.

By "disperse dyes" in this application I mean water-insoluble organic colors which are applied to the fiber from near-colloidal aqueous dispersion. Such dyes have hitherto been referred to generally as "acetate dyes"; see *The Chemistry of Synthetic Dyes and Pigments*, edited by H. A. Lubs (Reinhold Publishing Corp., 1955), pages 167 to 174.

A further object of this invention is to provide a dyeing process of the above type, using a carrier which is not toxic or malodorous. Other accomplishments and advantages of my invention will appear as the description proceeds.

Now according to my invention, the above objects are achieved by dyeing polyester fibers with disperse dyes in aqueous systems at about 100° C. using as carrier dimethyl terephthalate, and with the added condition that at least 2 parts of said carrier shall be present, in a state of solution, in each 1000 parts of dye bath.

To explain the significance of the last condition, I wish to state that dimethyl terephthalate is a solid, essen-

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tially insoluble in cold water. At higher temperatures, it may go sparingly into solution, but the rate of dissolution is so slow that even a concentration of 0.2% by weight, in the presence of a disperse dye, cannot be achieved in reasonable time, considering the economics of the dyeing process.

But according to my invention, the dimethyl terephthalate is added to the dye bath in a form of fine subdivision, corresponding at least to 100 mesh (U.S. sieve size series). Such a fine state of subdivision can be achieved, for instance, by conventional dry grinding in a ball mill or colloid mill. Alternatively, an aqueous dispersion of the agent may be milled in a suitable apparatus, and then evacuated to dryness, for instance, by spray drying. In either case, dispersing agents, anionic, cationic or non-ionic may be incorporated into the carrier during the milling procedure. When thus prepared, the product is a free flowing white powder, readily soluble in hot water to produce saturated solutions of the order of 1% concentration by weight.

Under these conditions, I find that the agent goes readily into solution and will dissolve to the extent of at least 0.2% by weight. Moreover, I find said concentration to be essentially the minimum effective for achieving good dyeings upon polyester fiber with disperse dyes. Aqueous baths in which the quantity of dimethyl terephthalate actually in solution is much below this figure, generally produce poor results.

As for quantities above this limit, such may be used up to and even exceeding the saturation point.

Since the dyeing is to be carried out essentially at 100° C., an open vessel may be employed. In other details, the dyeing may proceed in conventional manner, and may contain, for instance, dispersing agents, such as long-chain alcohol sulfates, or other anionic, cationic or non-ionic surface active agents.

My improved process yields dyeings of deep shades and characterized by good levelness and excellent light fastness. In addition to being useful for dyeing homogeneous fiber, my invention may be applied successfully to the dyeing of blends, i.e., fabrics composed of more than one material, for instance polyester fiber and a polyamide fiber. Heretofore, in the dyeing of such blends the disperse color would be sorbed preferentially by the polyamide portion of the fabric, since the rate of solution of the disperse dye in the polyamide fiber is considerably greater than its rate of solution in the polyester fiber. I found, to my surprise, that the use of dimethyl terephthalate effectively changes the solubility rate of the disperse dye in the polyester fiber.

Accordingly, with the use of dimethyl terephthalate as carrier, uniform dyeings of such blends may be obtained. This can be done, for instance, by first dyeing the blend using dimethyl terephthalate as carrier and thus causing the disperse color to be preferentially absorbed onto the polyester fiber. Then a second step is carried out which comprises dyeing with a nylon dye to match the shade of the polyester fiber, but in the absence of dimethyl terephthalate. Alternatively, simultaneous dyeing of the two fibers from a single bath may be achieved by incorporating in the dye bath an anionic nylon dye, in addition to the disperse dye and the novel carrier of this invention.

Without limiting my invention, the following examples are given to illustrate my preferred mode of procedure. Parts mentioned are by weight.

Example 1

A dye bath is prepared from 0.05 part of Disperse Yellow dye Prototype No. 534, 0.1 part of a commercial long-chain-alkyl sulfate mixture (C₁₄ to C₁₈), 0.5 part of dimethyl terephthalate whose particle size is not coarser

than 100 mesh, and 200 parts of water. A piece of spun polyethylene terephthalate (5 parts by weight) is boiled in this dye bath for 2 hours. The resulting dyeing is bright, level and has excellent light fastness.

When cellulose triacetate is dyed with the above dye bath under similar conditions, essentially the same results are obtained.

When the above two dyeing procedures are repeated except for using 0.2 part of dimethyl terephthalate instead of 0.5 part (i.e., 1 part of carrier per 1000 parts of dye bath instead of 2.5 parts per 1000) the dyeings obtained are much weaker than those obtained above.

Example 2

A dye bath comprising 0.05 parts of Disperse Red dye Prototype No. 370, 0.1 part of the long-chain-alkyl sulfate mixture mentioned in Example 1, 0.8 part of dimethyl terephthalate milled to a particle size not coarser than 100 mesh, and 200 parts of water is used to dye a 5 part sample of spun polyethylene terephthalate as in Example 1. The fabric is dyed a bright, level red shade which has excellent fastness to light and washing.

When cellulose triacetate is dyed with the above dye bath, similar results are obtained.

Example 3

A dye bath consisting of 0.05 part of Disperse Yellow dye Prototype No. 534, 0.1 part of long-chain-alkyl sulfate, 0.8 part of dimethyl terephthalate whose particle size is 10 mesh, and 200 parts of water is used to dye a 5 part piece of spun polyethylene terephthalate by boiling it in a dye bath solution for 2 hours. The dyeing obtained is very weak. This demonstrates the importance of initial particle size of the dimethyl terephthalate added.

When cellulose triacetate fabric is dyed with this dye bath similar results are obtained.

Example 4

A dye bath consisting of 0.05 part of Disperse Red dye Prototype No. 370, 0.1 part of the long-chain-alkyl sulfate of Example 1, 0.4 part of dimethyl terephthalate (particle size finer than 100 mesh) is prepared and is used to dye a 5 part piece of fabric composed of equal parts by weight of polyethylene terephthalate filament and nylon filament. The polyethylene terephthalate is dyed a strong, bright red shade; the nylon portion is only stained a light pink.

When the above procedure is repeated without the dimethyl terephthalate, the nylon is dyed a heavy red shade while the polyester fiber is only slightly dyed.

It will be understood that the details of the above examples may be varied within the skill of those engaged in this art. For instance, in lieu of the dyes named in the above examples, other disperse dyes may be used, for instance azo dyes, such as

Prototype No. 242—(yellow),
Prototype No. 245—(green shade of yellow),
Prototype No. 43—(orange),
Prototype No. 244—(red),
Prototype No. 236—(red);

disazo dyes, such as

SRA Golden Yellow XIII—(Lubs, page 171),
SRA Golden Orange I—(Lubs, page 171);

5 azomethine dyes, anthraquinone dyes, or any other of the disperse dyes discussed in the above cited Lubs text at pages 167 et seq.

I claim as my invention:

1. A process of dyeing polyester fiber of the group consisting of polyethylene-terephthalate and cellulose triacetate, which comprises treating the same at boiling temperature with an aqueous dispersion of a disperse dye in the presence of a carrier comprising dimethyl terephthalate, said dimethyl terephthalate being present in said aqueous treatment bath in quantity not less than 2 parts per 1000 by weight and being dissolved therein to an extent not less than 2 parts per 1000 by weight.

2. The process of preparing a dye bath for hydrophobic polyester fiber, which comprises dispersing a disperse dye in water at the boiling point, and adding to the hot aqueous dye bath thus produced solid dimethyl terephthalate in a state of subdivision sufficiently fine to dissolve in said dye bath and to produce therein a concentration not less than 0.2% by weight.

3. The process of preparing a dye bath for hydrophobic polyester fiber, which comprises dispersing a disperse dye in water at the boiling point, and adding to the hot aqueous dye bath thus produced solid dimethyl terephthalate in a state of subdivision not coarser than 100 mesh sieve, and in quantity sufficient to form a solution in said dye bath of a concentration not less than 0.2% by weight.

4. A process as in claim 1, wherein the fiber dyed is polyethylene terephthalate.

5. A process as in claim 1, wherein the fiber dyed is cellulose triacetate.

6. A process as in claim 1, wherein the fiber dyed is a blend of polyethylene terephthalate and polyamide fibers.

7. A process as in claim 1, wherein the fiber consists at least in part of polyethylene terephthalate.

8. A process of dyeing hydrophobic polyester fiber, which comprises treating the same at boiling temperature with an aqueous dispersion of a disperse dye in the presence of a carrier comprising dimethyl terephthalate, said dimethyl terephthalate being present in said aqueous treatment bath in quantity not less than 2 parts per 1000 by weight and being dissolved therein to an extent not less than 2 parts per 1000 by weight.

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