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PROCESS FOR DYEING POLYESTER FIBER

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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 fiber 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 we 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 our invention will appear as the description proceeds.

Now according to our invention, the above objects are achieved by dyeing polyester fibers with disperse dyes in aqueous systems at about 100° C. using as carrier a mixture of dimethyl terephthalate and benzanilide, in a ratio of from 1:3 to 3:1 by weight, and with the added condition that at least 2 parts of said mixed carrier shall be present, in a state of solution, in each 1000 parts of dye bath.

To clarify the significance of the last condition, we wish to explain that dimethyl terephthalate is a solid, essentially 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 our invention, the dimethyl terephthalate and benzanilide mixture 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 mixed agents may be milled in a suitable apparatus, and then evaporated 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 water to produce saturated solutions of the order of 2% concentration by weight. As already indicated, the proportion of each component in our binary carrier mixture is not less than 25% and not more than 75% by weight. The preferred compositions are those which contain the two components in about equal proportions by weight.

Under these conditions, we find that the mixed carrier goes readily into solution and will dissolve rapidly to the extent of at least 0.2% by weight. Moreover, we 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 said mixed carrier 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.

Our 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, our 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. We found, to our surprise that the use of our mixed carrier effectively changes the solubility rate of the disperse dye in the polyester fiber.

Accordingly, with the use of the above defined mixture of dimethyl terephthalate and benzanilide as carrier, uniform dyeings of such blends may be obtained. This can be done, for instance, by first dyeing the blend using our mixed 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 our mixed carrier. 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 our invention, the following examples are given to illustrate our preferred mode of procedure. Parts mentioned are by weight.

Example 1

A. 200 parts of dimethyl terephthalate and 50 parts of a magnesium lignosulfonate dispersing agent were ground in a micropulverizer to pass a 200 mesh screen.

B. 200 parts of benzanilide and 50 parts of a magnesium lignosulfonate dispersing agent were ground in a micropulverizer to pass a 200 mesh screen.

Products A and B were then combined and tumbled by a roller mill for two hours. The resulting mixture was an off-white free-flowing powder.

Example 2

A mixture of 6500 parts of dimethyl terephthalate, 6500 parts of benzanilide and 65 parts of the sodium salt of the alcohol sulfates obtained from a mixture of C_{12} to C_{18} alcohols was micropulverized to pass a 200 mesh screen. The resulting white powder was then tumbled on a roller mill for two hours to ensure thorough mixing of the material.

Example 3

A dye bath is prepared from 0.05 part of Disperse Red dye, Prototype No. 238, 0.1 part of a commercial long-chain alkyl sulfate mixture (C_{14} to C_{16}), 0.8 part of a mixture of equal parts by weight of dimethyl terephthalate and benzanilide which has first been reduced to a particle size finer 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 properties.

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

When the above procedures are repeated except for using the carrier mixture at an initial particle size of 10 mesh, the resulting dyeings are much weaker.

Example 4

A dye bath comprising 0.05 part of Disperse Red dye, Prototype No. 370, 0.1 part of long-chain alcohol sulfate (same as in Example 3), 0.8 part of a mixture of equal parts by weight of dimethyl terephthalate and benzanilide (particle size of the mixture less than 100 mesh), and 200 parts of water is used to dye a 5 part sample of spun polyethylene terephthalate as in Example 3. The fabric is dyed a bright, level red shade which has excellent fastness to light and wet treatments.

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

Example 5

Example 4 was repeated except for using as carrier a mixture of 0.2 part of benzanilide and 0.6 part of dimethyl terephthalate (state of subdivision, as in Example 4). The polyethylene terephthalate fabric was dyed a bright, level red shade, and the dyeing had excellent fastness properties.

When the same procedure was repeated with cellulose triacetate fabric, essentially the same results are obtained.

Example 6

Example 4 was repeated except for using as carrier a mixture of 0.6 part of benzanilide and 0.2 part of dimethyl terephthalate. The polyethylene terephthalate fabric was dyed a bright, level red shade, and it had excellent fastness properties.

When the same procedure was repeated using cellulose triacetate as the fabric, essentially the same results are obtained.

Example 7

When Example 3 is repeated using as dye Disperse Yellow, Prototype No. 534, a bright, level dyeing is obtained which has excellent fastness properties.

Example 8

Example 4 is repeated using a piece of fabric composed of equal parts by weight of polyethylene terephthalate filament and nylon. After dyeing at the boil for 2 hours, the polyethylene terephthalate portion is dyed a strong, bright red shade while the nylon is only stained a light pink.

When the experiment is repeated without any carrier, the polyester fiber is not appreciably dyed, while the nylon is dyed a heavy shade.

When using o-phenyl phenol as carrier the nylon is dyed with considerably more than ½ of the dye and it has a much heavier shade than the polyester fiber.

Example 9

5 parts by weight of a piece of fabric composed of equal parts by weight of polyethylene terephthalate fiber and cellulose acetate fiber (not cellulose triacetate) is dyed for 2 hours at the boil in a bath containing:

200 parts water
0.05 part Disperse Yellow dye, Prototype No. 534
0.1 part long-chain alcohol sulfate (C_{14} to C_{16})
0.8 part of a mixture of equal parts of dimethyl terephthalate and benzanilide (particle size finer than 100 mesh)

The polyethylene terephthalate portion of the fabric is dyed a strong, bright yellow shade, the cellulose acetate fiber is dyed a much weaker shade of yellow. This preferential dyeing of polyethylene terephthalate fiber makes possible the union dyeing of polyethylene terephthalate/cellulose acetate fabrics by subsequent dyeing of the acetate fiber with appropriate dyes at temperatures below 190° F. under which condition, the shade of the dyed polyethylene terephthalate is not appreciably changed.

Example 10

5 parts by weight of a piece of fabric composed of equal parts by weight of polyethylene terephthalate fiber, nylon fiber, and cellulose acetate fiber is dyed for two hours at the boil in a bath containing:

200 parts water,
0.05 part Disperse Red dye, Prototype No. 370,
0.1 part long-chain alcohol sulfate (C_{14} to C_{16}),
0.8 part of a mixture of equal parts of dimethyl terephthalate and benzanilide (particle size finer than 100 mesh)

The polyethylene terephthalate portion of the fabric is dyed a strong, red shade; the acetate is dyed a much weaker red shade; and the nylon is stained a light pink shade.

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)

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

We claim as our invention:

1. A carrier for applying disperse dyes to hydrophobic fiber, comprising a mixture of dimethyl terephthalate and benzanilide, each component being present in the mixture in proportion not less than 25% of the joint weight of the two, and said mixture being in powder form having an average particle size not coarser than 100 mesh sieve.

2. A carrier for applying disperse dyes to hydrophobic fiber, comprising a mixture of dimethyl terephthalate and benzanilide in essentially equal proportions by weight, said mixture being in powder form having an average particle size not coarser than 100 mesh sieve.

3. 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 a mixture of dimethyl terephthalate and benzanilide, each component of said mixture being present in proportion not less than 25% of the joint weight of the two, said carrier 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.

4. A process as in claim 3, wherein the fiber dyed consists at least in part of polyethylene terephthalate.

5. A process as in claim 3, wherein the fiber dyed is polyethylene terephthalate.

6. A process as in claim 3, wherein the fiber dyed is cellulose triacetate.

7. A process as in claim 3, wherein the fiber dyed is a blend of polyethylene terephthalate and polyamide fibers.

8. A process as in claim 3, wherein the fiber dyed is a blend of polyethylene terephthalate and cellulose acetate fibers.

9. A process as in claim 3, wherein the fiber dyed is a blend of polyethylene terephthalate, nylon and callulose acetate fibers.

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