The present invention relates to the dyeing of cellulose triacetate filamentary materials.

Cellulose triacetate filamentary materials are relatively slow dyeing as compared with conventional acetone-soluble secondary cellulose acetate. Moreover, when dyeing cellulose triacetate with disperse acetate dyes, the dyeings are not always level and washfast. If other fibers are present they will often be stained by the disperse acetate dye.

It is accordingly an object of the present invention to provide an improved process for dyeing cellulose triacetate.

Another object of the invention is to provide improved dyes of cellulose triacetate alone and/or in blending.

Other objects and advantages of the invention will become apparent from the following detailed description and claims wherein all parts are by weight unless otherwise specified and wherein all acetyl values are calculated as combined acetic acid by weight.

In accordance with one aspect of the present invention cellulose triacetate filamentary material is dyed in a dispersion of a disperse acetate dye in an aqueous liquor or dyebath having dissolved therein the acetic acid ester of triethyl citrate or β-butoxyethanol, i.e., acetyl triethyl citrate and β-butoxyethyl acetate. These esters are of low water solubility and are present in the dyebath in about 0.25 to 10% on the weight of the dyebath. If more ester is used than can be dissolved in the bath the excess will be dispersed and will be cleared when the fiber extracts the ester from the dyebath. For β-butoxyethyl acetate the preferred concentration is about 0.5 to 4% by weight of the dyebath and for acetyl triethyl citrate about 0.25 to 5%. For reasons of economy, quality of results and effectiveness at low concentration acetyl triethyl citrate is preferred.

The acetic acid esters employed in the indicated amounts effect increases in the rate and depth of dyeing, permitting desired shades to be obtained with only a fraction as much dye as is otherwise required and sometimes permitting deep shades to be obtained which cannot be obtained regardless of how much carrier is employed. In addition, the use of these esters effects an improvement in washfastness and hand, in strength retention following heat treatment and in safe ironing temperature. Finally, the esters permit knit fabrics such as tricot to be winch dyed without warp streaks which eliminates the need for more costly pressure equipment and very high temperatures. Filling bands are eliminated in woven fabrics having a cellulose triacetate filling and a warp of another fiber.

Dyeing may be carried out at temperatures in excess of about 50° C. up to temperatures beyond which the filamentary material is damaged and preferably is carried out at temperatures ranging from about 60 to 95° C. It may be carried out in pressure equipment, if desired, but as noted it can also be carried out on the winch or jig. Liquor ratios of 2:1 to 80:1 or more may be used although they preferably range from about 3:1 to 8:1 for jg dyeing and from about 20:1 to 50:1 for winch dyeing. Since the liquor ratio will govern the proportion of acetic acid ester to fiber at any given concentration of the ester, it is apparent that the optimum concentration will to some extent be related to the liquor ratio. Thus, at the preferred liquor ratios the preferred acetic acid ester, acetyl triethyl citrate is preferably used in a concentration of about 0.3 to 0.5% when dyeing on the winch and about 1 to 2% when dyeing on the jg. This corresponds to a range of about 3 to 25% of acetyl triethyl citrate based on the weight of fiber. At the preferred liquor ratios the β-butoxyethyl acetate is preferably used in a concentration of about 0.5 to 1% on the winch and about 2 to 4% on the jg, corresponding to about 6 to 25% on the weight of fiber.

The process is especially suited for dyeing with disperse acetate dyes such as Disperse Blue 27, Disperse Red 35, Disperse Yellow 37, and the like, although other classes of dyes such as basic dyes can be employed with somewhat lesser success.

As employed herein “cellulose triacetate” has reference to cellulose acetate having an acetyl value of at least about 59% and preferably at least about 61%, i.e. at most about 0.29 free hydroxyl group and preferably at most about 0.12 free hydroxyl group per anhydroglucose unit of the cellulose molecule.

The cellulose triacetate filamentary material may be in the form of staple fibers, tow, continuous filament or staple fiber yarns, fabrics and/or finished articles. They may be alone or in admixture with other fibrous materials such as wool, cotton, rayon, silk, nylon, glass, polymers and/or copolymers of vinylidene compounds such as acrylonitrile, ethylene, propylene, vinyl chloride, vinyl acetate, vinylidene cyanide, and the like. It is an advantage of the present invention that when dyeing a blend the blend fiber is not stained with the disperse acetate dye. If a mixture of dyes is employed, e.g. if a cellulose triacetate-wool blend is dyed with a bath containing one of the disclosed acetic acid esters along with a disperse acetate dye and an acid dye the cellulose triacetate will not be stained by the acid dye nor will the wool be stained by the disperse acetate dye.

Following dyeing the filamentary material may be rinsed and/or scoured in conventional manner to remove loose dye and to wash out the acetic acid ester. If a subsequent heat treatment is desired, notwithstanding the already increased safe ironing temperature, it may be carried out in conventional manner as by raising the temperature of the filamentary material above about 190° C. and preferably above 200° C. for a time sufficient to increase the crystallinity and to improve the washfastness but insufficient to produce any substantial damage. The heating can be effected by contact as with metal cans or rolls, by hot air as on an enclosed tenter frame or by radiant heat. Alternatively heat treatment can be carried out at somewhat lower temperatures by using steam at superatmospheric pressures.

The following examples further illustrate the invention.

Example 1

Tricot, knit of cellulose triacetate yarn which was sur-
3. A proven method for reducing the acetyl value is to saponify the fabric in a conventional manner. To achieve this, the acetyl value is reduced from 61.5 to 60.5%, followed by dyeing on a winch. The fabric is then cooled to 45°C in 15 minutes, followed by further treatment with water. Additional steps are taken to ensure the fabric is released from the winch. Dyeing is continued for 2½ hours, the bath is drained, and the fabric is rinsed with water while lowering the temperature slowly. The direct colors are then fixed with Sandofix WE, a cationic fixative by exhaustion at 60°C from a bath containing 0.5% on fabric weight, and the fabric is dried on a tenter frame. The fabric is then dyed with an almond color with no filling bands.

**Example IV**

The process of Example III is repeated with the following differences: the 3.6 kilograms of β-butoxyethyl acetate are replaced by 1.35 kilograms of acetyl triethyl citrate, the alkylarylpolyether alcohol is increased to 270 grams, and the temperature of the fabric is raised to 90°C. There are slightly more filling bands in Example III but far fewer than when dyeing with conventional carriers in the bath.

**Example V**

10 kilograms of the same fabric as in Example III is dyed on the jig with the same proportion of dyes as follows: the jig is filled with 125 liters of cold water and the fabric is wet. 1875 grams of acetyl triethyl citrate and 625 grams of an alkylarylpolyether alcohol are added over two passes of fabric through the bath and the fabric is run through twice more. The same dyes as in Example III are added in the same proportions over two passes of the fabric through the jig. After two more cold passes, the temperature is raised to 55°C over 35 minutes and maintained by steam injection. The fabric is passed through the bath for an hour, 5000 grams of sodium chloride are added and dyeing continued for 2½ hours. The bath is dropped, the jig is set to overflowing with water at 55°C and the fabric rinsed while allowing the temperature to drop. The fabric is treated with Sandofix WE, a cationic fixative, by exhaustion from a bath at 60°C containing 0.5% on fabric weight, extracted and dried. The fabric is a very light alendem color substantially free of filling bands. It is to be understood that the foregoing detailed description is given merely by way of illustration and that many variations may be made therein without departing from the spirit of our invention.

Having described our invention what we desire to secure is:

1. The process for dyeing textile material comprising cellulose trisacrate which comprises contacting said material at an elevated temperature with an aqueous dye-bath containing a disperse acetate dye and an acetic acid ester selected from the group consisting of acetyl triethyl citrate and β-butoxyethyl acetate present in about 3 to 25% of the weight of the textile material.

2. The process for dyeing textile material comprising cellulose trisacrate which comprises contacting said material at an elevated temperature with an aqueous dye-bath containing a disperse acetate dye and acetyl triethyl citrate present in about 3 to 25% of the weight of the textile material.

3. The process set forth in claim 2, wherein said acetyl triethyl citrate is employed in a concentration of about 0.25 to 5% on the weight of said dyebath.

4. The process for dyeing textile material comprising cellulose trisacrate which comprises contacting said material at an elevated temperature with an aqueous dyebath containing a disperse acetate dye and β-butoxyethyl acetate present in about 3 to 25% of the weight of the textile material.

5. The process set forth in claim 4, wherein said β-
butoxyethyl acetate is employed in a concentration of about 0.5 to 5% on the weight of said dyebath.

6. The process set forth in claim 1 including the further step of heat treating said material.

7. The process set forth in claim 6 wherein said material is scoured prior to said heat treatment.

8. The process set forth in claim 1 wherein said textile material is a warp knit fabric.

9. The process set forth in claim 1 wherein said textile material is a woven fabric and said filling consists essentially of cellulose triacetate.

10. The process set forth in claim 1 wherein said textile material comprises a combination of cellulose triacetate filamentary material and at least one other filamentary material.

11. The process set forth in claim 1 wherein said textile material also comprises cellulose and at least one cellulose dye is also present in said dyebath.

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