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SULFOLANYL ALKYL ETHERS IN DYEING CELLULOSE ACETATE, POLYVINYLIDINE CYANIDE AND GLYCOL-DICARBOXYLIC ACID POLYMER FILAMENTS

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The present invention relates to novel processes for dyeing synthetic filamentary materials and particularly to processes for dyeing selected synthetic filamentary materials with disperse dyes.

Disperse dyes were developed for the dyeing of secondary cellulose acetate, being applied from aqueous dispersion usually at elevated temperature. Take up of dye by the filamentary material is slow and incomplete. Where another filamentary material such as cotton or wool is present it is often stained, but not dyed, by the disperse dye especially where dyeing is carried out over prolonged periods with heat. Substances, called accelerants, have been found which will accelerate the take up of dye by the fiber and also increase the exhaustion. The reduced dyeing time minimizes the time during which a blend fiber is exposed to the dye and thus may diminish staining. On the other hand the accelerant may promote staining of the blend fiber and whether or not staining will occur is incapable of prediction, depending upon dyeing time, depth of shade, dyeing temperature, etc. In addition, certain accelerants by virtue of their volatility are of utility only when dyeing at low temperatures or under pressure.

The choice of an accelerant for a new filamentary material or the choice of a new accelerant for an old filamentary material is difficult since accelerants for one filamentary material may not function satisfactorily on another. Moreover, prediction is rendered impossible because the exact mechanism by which an accelerant functions is not understood. While it has been postulated that the accelerant swells the filamentary material to be dyed numerous known swelling agents are unsuited as accelerants either because they fail to speed up dyeing, because they cause staining of blend fibers, because they damage the principal or blend fiber, etc.

It is accordingly an object of the present invention to provide accelerants for dyeing nytril, polyester, cellulose triacetate and secondary cellulose acetate filamentary materials with disperse dyes.

A further object of the invention is to provide accelerant dyeing processes for dyeing blends of the aforementioned filamentary materials with other filamentary materials.

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.

In accordance with one aspect of the present invention dyeing of a filamentary material selected from the group consisting of nytril, polyester, cellulose triacetate and secondary cellulose acetate filamentary materials is carried out with a dispersion in water of a disperse dye and a non-volatile accelerant selected from the group consisting of an alkyl sulfolanyl ether and a substituted alkyl benzoate, the dyebath being applied by immersion, spraying, padding, etc. The accelerants boil in excess of about 175° C. and thus are suited for dyeing without pressure at high temperatures in excess of about 95° C., although lower temperatures may also be employed.

Representative sulfolanyl ethers which may be employed in accordance with the invention include the ethers of 3-sulfolanyl alcohol with an alkanol preferably containing about four to ten carbon atoms, e.g., butyl 3-sulfolanyl ether, isooctyl 3-sulfolanyl ether, hydroxyethoxyethyl 3-sulfolanyl ether, ethoxypropyl 3-sulfolanyl ether, and the like. Ethers of substituted sulfolanyl alcohols and sulfolanyl alcohols with the hydroxyl group in 2-position can also be used.

Advantageously the substituted alkyl benzoates which may be employed in accordance with the invention have a total of about 9 to 12 carbon atoms and preferably the substituents are lower alkyl or lower alkoxy radicals attached either to the benzene ring or to the alkyl ester group, e.g., methyl p-toluate, methoxyethyl p-toluate, methoxyethyl benzoate, and the like.

The accelerants are generally employed in about 2 to 20% and preferably about 5 to 12% on the weight of fiber. Smaller amounts generally do not accelerate dyeing sufficiently and larger amounts are uneconomical. The concentration of accelerant in the dyebath may vary within wide limits, e.g., as low as about 0.1% when winch dyeing or as high as about 6% when jig dyeing although even higher or lower concentrations can be tolerated. Advantageously there is also present in the dyebath about 0.02 to 0.2 and preferably about 0.04 to 0.1% of an emulsifying agent, e.g., an anionic agent such as sodium dodecylbenzene sulfonate or a nonionic agent such as an alkarylpolyether alcohol. This serves to maintain in dispersed condition the accelerant when the latter is present in excess of its solubility and it also maintains in dispersion the dyestuff which is generally present in about 1 to 10% on fabric weight depending upon its color and the desired shade. Other ingredients conventional in the dyeing art such as thickeners, humectants, or the like can also be present in the dyebath especially when printing. The temperature and duration of dyeing will depend upon each other as well as upon the identity of the filamentary material, the dye and the accelerant and upon the depth of shade desired. Generally, the temperature of the dyebath ranges between about 50° C. and the boil and most frequently between about 90° C. and 100° C. Pressure equipment will permit use of temperatures above the normal boiling point of the dyebath and will permit a particular effect to be achieved with a smaller proportion of accelerant.

In accordance with another aspect of the invention it has been found that the use of p-methyl toluate as an accelerant for dyeing nytril filamentary material is particularly efficacious resulting in full shades, high exhaustion and rapid dyeing. Other accelerants heretofore employed with other fibers do not yield equally satisfactory results.

The recited filamentary materials may be dyed in blends with one another in which event more or less uniform coloration, i.e., union dyeing, will be achieved. The recited filamentary materials may also be dyed in blends with other filamentary materials to which disperse dyes are non-substantive such as wool, cotton, rayon, or the like, in which event a reserve effect will be achieved. It is an advantage of the present invention that little or no staining of such other filamentary material occurs and whatever slight staining occurs can readily be cleared. It is also possible to include in the dyebath dyes substantive to the other filamentary materials so as to obtain a cross-dyed effect.

The filamentary materials dyed in accordance with the invention may be in the form of garments or other finished articles, knit, woven or non-woven fabrics, filament or spun yarn, tow, roving, sliver, staple fiber, or the like.

As employed herein, nytril refers to filamentary materials comprising polymers of vinylidene cyanide. These may be homopolymers or advantageously copolymers with one or more monomers such as vinyl acetate, styrene, or the like. A preferred material is a copolymer comprising about 45 to 55 mol percent of vinylidene cyanide, e.g.,

the vinylidene cyanide and vinyl acetate form a copolymer in which the different monomer units are alternated in regular manner as in the product sold under the trademark "Darvan." Polyester refers to linear superpolyesters such as the condensation products of ethylene glycol, polyethylene glycol and/or another glycol with terephthalic acid and/or other dicarboxylic acids such as adipic or sebacic acid, e.g., polyethylene terephthalate, such as is sold under the trademark "Fortrel."

Cellulose triacetate refers to cellulose acetate having an acetyl value in excess of about 59% and preferably in excess of about 61%. Secondary cellulose acetate has reference to acetone soluble cellulose acetate, i.e., cellulose acetate having an acetyl value of about 54 to 58% by weight calculated as combined acetic acid.

The filamentary materials may contain other chemical entities either in the polymer chain or as additives cospun therewith or subsequently applied for the purpose of varying the physical and/or chemical properties such as dye receptivity.

The invention will be further described in the following illustrative examples.

Example I

(*a*) 10 parts by weight of 3 denier staple fibers of a copolymer comprising alternating units of vinylidene cyanide and vinyl acetate (sold under the trademark "Darvan") are immersed in 300 parts of water at 50° C. and containing 1.2 parts of methyl p-toluate and 0.3 part of sodium dodecylbenzenesulfonate. After 10 minutes of agitation, 0.3 part of the disperse dye 1-methylamino-4-β-hydroxyethylaminoanthraquinone (Disperse Blue 3) is added, the bath temperature is raised to 95° C. over 30 minutes. After two hours of agitation, the bath is dropped, the fiber is rinsed for 5 minutes in cold running water, scoured for 20 minutes at 80° C. with a 0.1% soap solution, rinsed again in cold running water and dried. The dyed fabric is fast to light as well as to washing and dry cleaning solvents, indicating that the dye is in the fiber and not just held on the surface. The shade is full, the dye being approximately 65% exhausted from the bath.

(*b*) A control run, identical except for omission of the methyl p-toluate, resulted in a pastel shade and only about 10% exhaustion of the dye from the bath.

(*c*) Substituting o-phenylphenol for the methyl p-toluate, the color value and dye exhaustion are almost 20% less than (*a*).

Example II

The process of Example I is repeated except that the fiber is cellulose triacetate having an acetyl value of 61.9%. The fiber is dyed a full shade with 70% exhaustion of the dyebath. Without accelerant only 40% exhaustion is obtained.

Example III

The process of Example I is repeated on fibers of polyethylene terephthalate. 60% exhaustion of the dyebath is acchieved compared with only 10% in the absence of accelerant.

Example IV

The process of Example I is repeated except that the methyl p-toluate and the sodium dodecylbenzenesulfonate are respectively replaced by isoctyl 3-sulfolanyl ether and an alkylarylpolyether alcohol sold under the trademark Triton X102. Substantially the same results are achieved as in Example I.

Example V

The process of Example IV is repeated except that the fiber is cellulose triacetate having an acetyl value of 61.9% and the dye is a prototype of Disperse Blue 27.

Example VI

The process of Example V is repeated employing Disperse Red 35 as the dye and replacing the fiber by 10 parts of a check fabric woven of alternate warp and filling bands of wool and of cellulose triacetate having an acetyl value of 61.9%. The cellulose triacetate is dyed a deep shade and the wool is stained so slightly that it could be dyed even without a pre-scour; as a matter of fact a wool dye could be incorporated into the same dyebath since it is not necessary to remove stain from the wool. The relative absence of stain permits the wool to be dyed lighter shades and it renders the dyeing more washfast and light fast since if the wool had picked up disperse dye it would give it up when not desired. In addition the absence of wool stain makes it easier to match shades.

As contrasted with o-phenylphenol or other carriers heretofore employed the depth of shade on the cellulose triacetate is greater and the wool staining is appreciably less while the exhaustion is at least equal.

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 by Letters Patent is:

1. The process which comprises immersing a filamentary material selected from the group consisting of vinylidine cyanide polymer, polyester condensation product of a glycol and a dicarboxylic acid, cellulose triacetate and secondary cellulose acetate filamentary materials in an aqueous dispersion of a disperse dye containing about 0.1 to 6% of an alkyl sulfolanyl ether present in an amount ranging from about 2 to 20% of the weight of said filamentary material.

2. The process set forth in claim 1, wherein said alkyl sulfolanyl ether is the ether of 3-sulfolanyl alcohol with an alkanol having about four to ten carbon atoms.

3. The process which comprises immersing a filamentary material of a polyester condensation product of a glycol and a dicarboxylic acid, in an aqueous dispersion of a disperse dye containing about 0.1 to 6% of the ether of sulfolanyl alcohol with an alkanol having about four to ten carbon atoms, said ether being present in about 2 to 20% of the weight of said filamentary material.

4. The process which comprises immersing a cellulose triacetate filamentary material in an aqueous dispersion of a disperse dye containing about 0.1 to 6% of the ether of sulfolanyl alcohol with an alkanol having about four to ten carbon atoms, said ether being present in about 2 to 20% of the weight of said filamentary material.

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