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[54] **DYEING COTTON-POLYESTER TEXTILES WITH ONE-BATH MIXTURE OF DISPERSED AND CELLULOSE-REACTIVE DYESTUFF**

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[51] **Int. Cl.** **D06p 3/82**

[58] **Field of Search** 8/21, 25, 26

[56] **References Cited**

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[57] **ABSTRACT**

A process for dyeing polyester-cellulose union materials in a single bath containing a mixture of disperse and specified cellulose-reactive dyes by heating to a temperature of 100°–140° C and pH 5.0 to 5.5 until the polyester component is dyed then cooling to 80° C, adding alkali and a neutral salt if the latter is not already present, and dyeing at 80°–90° C to fix the reactive dye.

The reactive dyes used are those which when dyed on cotton at 120° C for 45 minutes give an equal or only a marginally less depth of shade than a similar dyeing carried out at 80° C.

2 Claims, No Drawings

DYEING COTTON-POLYESTER TEXTILES WITH ONE-BATH MIXTURE OF DISPERSED AND CELLULOSE-REACTIVE DYESTUFF

This invention relates to a new dyeing process and more particularly to a new process for dyeing union textile materials of cellulose and polyester with, respectively, cellulose-reactive and disperse dyestuffs.

A number of methods for dyeing union materials of this kind with such dyestuffs are known. In some cases, the method involves a continuous processing method whereby the material to be dyed is padded in a mixture of dyestuffs and then the latter are fixed on the textile material by a heat treatment. This method is subject to the disadvantage that it can only be carried out in the specialized, expensive machines developed for continuous processing.

Other methods are known in which the textile material is dyed in two separate dye liquors, one containing the reactive dye and the other the disperse dye. While these methods are more convenient in that they can be carried out in cheaper and more readily available machinery, they have the disadvantage that two different dyeing vessels are required or that there is a considerable loss of time in replacing one dye liquor by the other in the dyeing vessel.

Thus, there is a need for a convenient, "one-pot" dyeing method in which the material can be treated with a single dye liquor containing both kinds of dyestuff with a minimum of additions during the process. Processes of this kind have been described previously, e.g., in U.S. Pat. Nos. 1,070,928, 1,129,440 and 1,133,650. However these processes involve use of an alkaline dyebath at temperatures of 95° C or above and the number of disperse dyestuffs which will withstand such conditions without aggregating is very small.

It has now been found that a large number of the commercially-available cellulose-reactive dyestuffs are sufficiently stable to resist hydrolysis in a weakly acid dyebath at temperatures of from 100° C to 140° C for dyeing of the polyester component to take place in the presence of both kinds of dyestuffs, the reactive dye thereafter being fixed by addition of alkali to the bath. A simple test for suitability of the reactive dyestuff is as follows:

Two dyeings are carried out on cotton hanks using equal amounts of the dyestuff. In each case the dyebath contains also 80 g/l of common salt and 5 g/l of sodium-m-nitrobenzene sulphonate. In the first of such dyeings, identified as method (*a*), dyeing is carried out for 45 minutes at 120° C at pH 5.0–5.5 (0.15 g/l of acetic acid). Then, the liquor is cooled to 80° C, sodium carbonate is added (20 g/l) and the dyeing is continued for 45 minutes. In the second of such dyeings, identified as method (*b*), dyeing is carried out for 45 minutes at 80° C under neutral conditions. Then sodium carbonate is added (20 g/l) and the dyeing is continued for 45 minutes. Both hanks are then rinsed in water and secured at the boil in 0.3% detergent for 15 minutes to remove loose dyestuff.

In many cases it will be found that the two dyeings are of equal strength. Where, however, the dyeing produced by method (*a*) is weaker than that produced by method (*b*), the difference can be visually estimated using a geometric grey scale card as used for the effect of washing tests on dyeings, B.S.2663:1961. A dyestuff is considered suitable if the difference in depth is no greater than a grading of 4 on this card. However, a reactive dyestuff containing the dichloro- or dibromo-s-triazine group is too unstable, even at 80° C, to be suitable for use in this new process.

The following cellulose-reactive dyestuffs, referred to by their Colour Index (Reactive) reference numbers have, for example, been found to pass this test:

Black — 8
Brown — 2, 7, 9, 17 and 19
Blue — 2, 3, 5, 19, 21, 25, 26, 40, 46, 49, 64 and 71
Green — 5 and 8
Orange — 2, 7, 12, 13 and 35
Red — 3, 13, 19, 56 and 58
Yellow — 3, 6, 15 and 18.

Accordingly, the invention provides a process for dyeing cellulose-polyester union textile materials by a one-bath method wherein the material is treated with a dye liquor containing a mixture of a disperse dyestuff and a suitable (as hereinbefore defined) cellulose-reactive dyestuff at pH 5.0–5.5 and a temperature of 100° to 140° C until the polyester component has been dyed with the disperse dye. Thereafter the dye liquor is cooled to 80°C, rendered alkaline and dyeing is continued at 80°–90° C at a pH greater than 8.0 until the cellulose component has been dyed with the cellulose-reactive dye, at least the second step being carried out in the presence of a neutral salt to assist exhaustion of the cellulose-reactive dye on to the textile material.

With the exception of dyes the dibromo- or dichloro-s-triazine group as the cellulose-reactive group, a large number of the cellulose-reactive dyes commercially available at the present time are suitable for use in the new process. Among those suitable are, for example, dyes containing di- and tri-chloropyrimidine, sulphatoethyl sulphone, dichloroquinoxaline, β-chloroethylaminosulphonyl and, more especially mono-chloro-s-triazine groups containing amino, etherified hydroxy or anilino or substituted anilino groups. The dyes include members of the phthalocyanine, anthraquinone and azo, including monoazo, disazo and metal complex azo series.

The disperse dyes used can belong, for example, to the azo, anthraquinone, nitro, styryl or quinaphthalone series. They form stable dispersions in the dye liquor and possess good affinity for polyester fibres and good light fastness on the fiber. They are free from strongly ionizing groups such as SO_3H and CO_2H and from groups which are readily converted into strongly ionizing groups under the dyeing conditions.

The new process can conveniently be carried out by the following procedure. First there is formed a weakly acid dye liquor containing both kinds of dyestuffs. The acidity is preferably brought about by adding a water-soluble organic acid, e.g., acetic acid. The dye liquor preferably contains a small amount of a dispersing agent and of a weak oxidizing agent, e.g., sodium-m-nitrobenzene sulphonate. A neutral electrolyte can be added at this stage, but is preferably added at a later stage after dyeing at 100° to 140° C is complete. After entering the union textile material, the dye liquor is heated to dyeing temperature, i.e., 100° to 140° C and maintained at this temperature until the polyester component of the union has been dyed.

At this point the dye liquor is cooled to 80° C and, if sufficient neutral electrolyte is not already present, more is added. In this case the dyeing is continued for a period of time, e.g., for 15 minutes, for partial ex-

haustion of the reactive dye to take place. Thereafter the alkali is added and dyeing is continued until fixation of the reactive dye has been achieved. This will normally take from 30 to 90 minutes.

As dispersing agents there may be used any anionic or non-ionic kinds commonly used for dye liquors containing disperse dyestuffs. as specific examples of these, there may be mentioned disodium methylene naphthalene disulphonate, sodium salts of cresol sulphonate/urea formaldehyde condensates and polyethanoxy condensates of aliphatic and aromatic residues, for example of amines, alcohols and phenols. The amount of dispersing agent used may be, for example, from 1 to 5 g/l.

As neutral electrolytes which can be used, there may be mentioned sodium chloride and sodium sulphate. For the first stage of the process, carried out above 100° C, it is desirable to use a maximum of 50 g of the electrolyte per liter of dye liquor, and preferably to carry out this stage in the absence of added electrolyte. However in the second stage of the process, carried out at from 80° to 90° C, electrolyte must be present and, dependent on the cellulose-reactive dyestuff being used, a higher concentration of electrolyte may be desirable, for example, up to 100 grams of the electrolyte per liter of dye liquor.

For making the dye liquor alkaline, a wide range of alkaline, but preferably non-caustic, materials may be used, e.g., metal salts of weak inorganic or organic acids, e.g., the hydroxides, bicarbonates, carbonates, silicates and phosphates of sodium or potassium.

On completion of dyeing, the textile material may be rinsed and subjected to the usual washing treatments conventional in the art for materials dyed with reactive dyestuffs, i.e., including a scouring step with a weak (e.g. 0.3%)solution of a detergent to remove unfixed dyestuff.

The new process has the advantage that it can be carried out in the conventional pressurized dye vessels commonly used for dyeing with disperse dyestuffs and gives a procedure in which only one dye liquor is used. In the preferred process, omitting the neutral electrolyte while dyeing the polyester component of the union, the chances of aggregation are reduced to a minimum so that the process is extremely convenient for the package dyeing of materials or the dyeing of tightly woven or bulky materials which are extremely suceptible to uneven dyeing if any aggregation takes place.

The invention is illustrated but not limited by the following examples in which parts are by weight:

Example 1

1000 parts of a dye liquor are made up containing 3 parts of disperse dye (see below), 1 part of a dispersing agent, sodium salt of cresol sulphonate/urea-formaldehyde condensate, sold under the trademark Dispersol BD, 0.15 part of acetic acid, 5 parts of sodium m-nitrobenzene sulphonate, 50 parts of sodium sulphate and 4 parts of reactive dye (see below). 100 parts of a 67:33 polyester-cotton yarn were entered at 60° C and the dye-liquor was heated to 130° C during 30 minutes and kept at this temperature for 1 hour.

The dye liquor is then cooled to 80° C, 20 parts of sodium carbonate (anhydrous) are added and the treatment is continued at 80° C for 1 hour.

The yarn is then removed from the dye liquor, rinsed in water, scoured in a 0.3% aqueous solution of detergent for 15 minutes at the boil, rinsed again in water and dried.

Using as the disperse dye, the product obtained by coupling diazotized 6-bromo-2,4-dinitroaniline with 5-acetylamino-2-methoxy-N-(β′-methoxy-β-ethoxycarbonylethyl)aniline and as the reactive dye, the product obtained by condensing cyanuric chloride with the 1:1 copper complex of 2-methylamino-6-[4′-(2″,5″-disulphophenylazo)-5′-methyl-2′-hydroxyphenylazo]-5-naphthol-7-sulphonic acid (1 mole) and ammonia (1 mole), a deep, even, navy-blue dyeing is obtained.

Example 2

A dye liquor is made up as in Example 1 except that the sodium sulphate is omitted. Dyeing is then carried out at 130° C as in Example 1, and the liquor is cooled to 80° C. Then 100 parts of sodium chloride are added, followed after 10 minutes by 20 parts of sodium carbonate. Dyeing is continued for 50 minutes, then the yarn is washed as in Example 1.

A similar result is obtained.

What we claim is:

1. A process for dyeing cotton-polyester union textile materials by a one-bath method comprising

a. dyeing said material in said bath comprising a dye liquor containing a mixture of a disperse dyestuff and a cellulose-reactive dyestuff at a temperature of 100° to 140° C, said dye liquors having a pH of 5.0–5.5, for a time sufficient to dye the polyester component of said textile material with said disperse dyestuff

b. cooling said dye liquor to 80° C

c. adding sufficient alkali to said dye liquor so that the pH thereof is greater than 8.0

d. continuing dyeing said textile material at a temperature of 80°–90° C in the presence of a neutral electrolyte for a time sufficient to dye the cotton component of said textile material with said reactive dyestuff,

said cellulose-reactive dyestuff being such that the difference in depth of dye shade between (1) a cotton textile material dyed in accordance with method (*a*) at 120° C with the said cellulose-reactive dyestuff and (2) the said cotton textile materials dyed in accordance with method (*b*) at 80° C with the said cellulose reactive dyestuff is no greater than a grading of 4 on the geometric grey scale card of B.S. 2663:1961, the dyeing of the said cotton material in accordance with method (*a*) being carried out in an aqueous saline bath for 45 minutes at a pH of 5.0–5.5 with (*i*) subsequent cooling to 80° C, (*ii*) addition of 20 g/l of sodium carbonate and (*iii*) continued dyeing at 80° C for 45 minutes, and the dyeing of the said cotton material in accordance with method (*b*) being carried out in an aqueous saline bath for 45 minutes under neutral conditions with (*i*) subsequent addition of 20 g/l of sodium carbonate and (*ii*) continued dyeing at 80° C for 45 minutes.

2. The process of claim **1** wherein the neutral electrolyte is selected from the group consisting of sodium chloride and sodium sulphate.

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