

[54] **PROCESS FOR THE DYEING OF
TEXTILE MATERIAL OF MIXTURES
OF POLYESTER FIBERS AND
CELLULOSE FIBERS**

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[56]

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[57]

ABSTRACT

Process for the dyeing of textile materials consisting of mixtures of polyester fibers with cellulose fibers, wherein the said textile materials are treated with an alkaline solution which contains a coupling component, a disperse dyestuff and wetting or dispersing agents, dried and subsequently treated with an acid solution, which contains, in addition to compounds having an acid reaction, a diazotized aromatic or heterocyclic amine, dried and then subjected to a heat treatment.

7 Claims, No Drawings

PROCESS FOR THE DYEING OF TEXTILE MATERIAL OF MIXTURES OF POLYESTER FIBERS AND CELLULOSE FIBERS

The present invention relates to a process for the dyeing of textile material of mixtures of polyester fibers and cellulose fibers.

It is known that textile materials consisting of mixtures of polyester fibers and cellulose fibers can be dyed fast shades using disperse dyestuffs and water-insoluble azo dyestuffs produced on the fiber. This process is effected in such a manner that first the polyester fiber portion is dyed with disperse dyestuffs at boiling temperature in the presence of dyeing accelerators, or under high temperature dyeing conditions or according to the so-called thermosol process, and then the water-insoluble azo dyestuff is produced on the cellulose fiber portion by impregnation with a coupling component and following development with a diazotized aromatic amine. This method of operation yields dyeings of good quality. It is, however, very time-consuming, since the disperse dyestuff, the coupling component and the diazotized aromatic amine have to be applied in separate baths.

Now we have found that this disadvantage in the dyeing of textile material consisting of mixtures of polyester fibers and cellulose fibers can be avoided by treating the textile material with alkaline solutions which contain a coupling component, a disperse dyestuff as well as wetting or dispersing agents, then drying them and treating them subsequently with an acid solution which contains, in addition to compounds having an acid reaction, a diazotized aromatic or heterocyclic amine, then drying them and subjecting them to a heat treatment.

The process of the present invention is effected by impregnating the textile material, for example a mixed fabric consisting of polyester and cellulosic fibers, with an alkaline solution which contains a coupling component, a disperse dyestuff as well as a wetting or dispersing agent, and then drying the material. For this purpose, it is suitable to use a predrying tunnel after the impregnating foulard in order to avoid migration of the disperse dyestuff during drying and formation of unlevel parts by contact with the rolls. The alkali content of the impregnation bath shall be so high that the coupling component does not precipitate. Depending on the nature and concentration of the coupling component, about 5 - 30 ml. of 32.5 percent sodium hydroxide solution are used as alkali per one liter of water. After drying, the textile material is impregnated on a foulard or a nip-pad dyeing apparatus with a solution which contains a diazotized aromatic or heterocyclic amine as well as a dispersing agent and such a quantity of a substance having an acid reaction that the material to be dyed has a pH in the range of from 4 to 7, after the passage or after the nip-padding. In this pH-range the coupling component on the material couples with the diazotized amine, whereby the dyestuff is formed on the cellulose fiber portion of the mixed fabric. The following drying process is suitably effected again in a drying tunnel, because the disperse dyestuff on the goods is not yet fixed and migration of this dyestuff portion or any influence effected by the guide rolls should be avoided to obtain level appearance of the goods. After the drying process, the goods are

treated for about 30 seconds to 2 minutes on a thermosoling apparatus at temperatures in the range of from about 170°-210° C. During this time the disperse dyestuff and a part of the water-insoluble azo dyestuff that has separated in the dye-bath and has been absorbed by the fiber are fixed on the polyester portion of the mixed fabric. The heat treatment can be carried out with hot air or by contact heat. The adjustment of the pH-value to about pH 4 to 7 on the goods in the treatment with the acid solution of the diazotized amine is necessary in order to avoid damage of the cellulose fiber portion of the mixed fabric during the treatment with dry heat by too low a pH-value and, on the other hand, also in order to prevent the disperse dyestuff from being hindered by an alkaline medium to absorb evenly on the polyester fiber portion during the heat treatment. Finally, the fabric is washed with an alkaline solution, rinsed and dried. The water-insoluble azo dyestuff is obtained on the cellulose fiber portion in full yield and clearness. The dyeings so produced have very good fastness properties.

Accordingly, in the process of the invention, the formation of the water-insoluble azo dyestuff on the cellulose fiber portion takes place when the goods, that have been impregnated with the coupling component and the disperse dyestuff, are introduced into the acid development bath in the presence of the disperse dyestuff, without the latter being at first fixed on the polyester fiber portion. The fixation of the disperse dyestuff on the polyester fiber portion is effected in the following heat treatment at temperatures in the range of from 170°-210° C.

As textile material, mixtures may be used which contain from 25 percent of polyester fibers and 75 percent of cellulose fiber to 70 percent of polyester fibers and 30 percent of cellulose fibers.

As polyester fibers, those made of aromatic polyesters, for example of terephthalic acid or diphenyl-4,4'-dicarboxylic acid and alkanediols or 1,4-cyclohexanedimethanol, as well as of triacetylcellulose are suitable. As cellulose fibers, natural and regenerated cellulose fibers may be used.

As coupling components, especially aromatic o-hydroxycarboxylic acid arylamides or acylacetic acid arylamides may be used in the process of the invention, for example, 2,3-hydroxynaphthoic acid arylamides, 6-bromo- or 6-alkoxy-2, -3-hydroxynaphthoic acid arylamides or acetoacetic acid arylamides, which have a low to medium substantivity towards cellulose fibers. If suitable impregnating apparatus are used, for example such with a small-sized liquor container, there may also be used coupling components which have a high substantivity towards the cellulose fibers, for example the condensation products of 2,3-hydroxynaphthoic acid with polynuclear isocyclic or heterocyclic amines such as aminonaphthalenes, aminocarbazoles or aminodiphenylene-oxides, furthermore heterocyclic o-hydroxycarboxylic acid arylamides, for example 5-hydroxy-1,2,1',2'-benzocarbazole-4-carboxylic acid arylamides, 2-hydroxy-carbazole-1 or -3-carboxylic acid arylamides as well as 4,4'-bis-acetoacetylaminodiphenyls or 2-hydroxyanthracene-3-carboxylic acid arylamides.

As diazotized primary aromatic or heterocyclic amines mono-or diamines, for example diazotized

chloroanilines, dichloro-anilines, chloro-toluidines, chloro-anisidines, nitranilines, nitrotoluidines, nitroanisidines, nitroxylidines, nitrophenetidines, cyanotoluidines, cyanoanisidines, amino benzene-sulfonic acid amides, aminobenzene-carboxylic acid amides, aminophenylalkyl-, -aryl- or -aralkyl-sulfones, aminodiphenylethers, trifluoromethyl-anilines, monoacylated phenylenediamines, aminoazobenzenes or 4-aminodiphenylamines as well as tetrazotized 4,4'-diaminodiphenyls may be used.

As wetting or dispersing agents in the alkaline impregnation bath, condensation products of higher molecular fatty acids and protein degradation products, condensation products of higher molecular fatty acids and aminoalkylsulfonic acids, condensation products of formaldehyde and naphthalene-sulfonic acids as well as purified sulfite cellulose waste liquor may be used.

As compounds having an acid reaction, organic acids, for example, acetic acid, formic acid, propionic acid, lactic acid, glycolic acid, tartaric acid or citric acid, or salts having an acid reaction, for example sodium mono-phosphate, aluminum sulfate, or zinc sulfate may be used. If non-volatile acid compounds are used, care must be taken not to use a higher quantity than is necessary for the neutralization of the alkali which is on the goods, in order to prevent a damage of the cellulose fiber portion during the following heat treatment.

As disperse dyestuffs, such dyestuffs may be used which on account of their thermal properties can be used in the so-called thermosol process, i.e., dyestuffs which dye polyester fibers at temperatures in the range of from about 170° to 210° C and which do not soil the dyeing apparatus due to their high volatility. The dyestuffs must be stable against the lye present in the impregnation bath, for example, they must not tend to coagulation during the about two hours' stay in the impregnation bath. Suitable disperse dyestuffs of the azo and anthraquinone series are described in the Color-Index, second edition 1956, vol. 1, pages 1659-1742 and Supplement 1963, pages S 179-224 as well as in the corresponding Additions and Amendments, No. 1 (September 1963) to No. 22 (January 1969).

The process of the invention may be executed in continuous as well as in semi-continuous manner. In contradistinction to the known method, wherein the disperse dyestuff is fixed in a first stage on the polyester fiber portion of the mixed fabric by a thermosol process, the process of the invention allows to combine

the application of the disperse dyestuff with the preparation of the water-insoluble azo dyestuff on the cellulose fiber portion of the mixed fabric and to save thereby one stage of operation. The following example illustrates the invention:

EXAMPLE

A mixed fabric consisting of 67 percent of polyethylene glycol terephthalate fibers and 33 percent of cotton was impregnated with a solution containing, per liter of water, 16.6g of 2,3-hydroxynaphthoylaminobenzene, dissolved in 16.6ml of 32.5 percent sodium hydroxide solution and 90° C hot water, as well as 4g of a condensation product of formaldehyde and β -naphthalene-sulfonic acid, 0.5 g of the sodium salt of 2,5-dibutyl-naphthalene-sulfonic acid and 30 g of C.I. Disperse Red 90.

The fabric was then squeezed to a bath absorption of 60 percent of the weight of the fabric, dried and impregnated with a solution containing, per liter of water, 10.6 g of diazotized 1-amino-2-methyl-4-chlorobenzene, 21 ml of 50 percent acetic acid, 20 g of sodium acetate and 2 g of a reaction product of about 20 mols of ethylene oxide and 1 mol of octadecylalcohol. The fabric was then squeezed to a bath absorption of 60 percent of the weight of the fabric and conducted through a drying apparatus in such a manner into a thermosoling chamber or over hot rolls that the fabric was heated for 30-60 seconds to a temperature in the range of from 180° to 210° C. Finally, the fabric was treated at boiling temperature with a solution containing, per liter of water, 2 g of a 30 percent reaction product of about 10 mols of ethylene oxide and 1 mol of nonylphenol, and 2 ml of a 25 percent solution of the sodium salt of nitrilo-triacetic acid, and the fabric was then rinsed hot and cold. The after-treatment of the dyeing can also be effected at 60° C or 95° in a bath which contains per liter of water, 3 g of a mixture of 63 percent of perchloroethylene, 17 percent of a reaction product of about 10 mols of ethylene oxide and 1 mol of nonylphenol and 20 percent at isopropyl alcohol, and 2 g of a 25 percent solution of the sodium salt of nitrilo-triacetic acid. A full red dyeing having good fastness properties was obtained.

The following table indicates further coupling components, diazotized amines as well as disperse dyestuffs which may be used according to the invention and the shades which can be obtained on mixed fabrics consisting of polyethylene glycol terephthalate fibers and fibers of cotton or regenerated cellulose:

| Coupling component | Disperse dyestuff | Diazotized amine | Shade |
|---|--|--|----------------|
| 2,3-hydroxynaphthoylaminobenzene | C.I. Disperse Red 90 | 1-amino-2-methyl-5-chlorobenzene | Yellowish red. |
| 1-(2,3'-hydroxynaphthoylamino)-2-methoxybenzene | do | do | Do. |
| 2,3-hydroxynaphthoyl-aminobenzene | do | 1-amino-2-nitro-4-chlorobenzene | Red. |
| Do | C.I. Disperse Red 82 | 1-amino-2-methoxy-4-nitrobenzene | Bluish red. |
| Do | C.I. Disperse Red 82 plus C.I. Disperse Blue 81 | 4-amino-3,2'-dimethyl-1,1'-azobenzene | Scarlet. |
| 1-(2'-hydroxycarbazole-3'-carboxyl-amino)-4-chlorobenzene | C.I. Disperse Orange 13 plus C.I. Disperse Blue 95 plus C.I. Disperse Red 82 | 1-amino-2-methyl-5-chlorobenzene | Brown. |
| 4,4'-bis-acetoacetyl-amino-3,3'-dimethyl-diphenyl | C.I. Disperse Yellow 64 | do | Yellow. |
| 2,3-hydroxynaphthoyl-aminobenzene | C.I. Disperse Blue 81 plus C.I. Disperse Orange 13 plus C.I. Disperse Red 82 | 4-amino-2,5-dimethoxy-4'-nitro-1,1'-azobenzene | Black. |
| 1-(2',3'-hydroxynaphthoylamino)-2-methoxybenzene | C.I. Disperse Blue 81 | 4,4'-diamino-3,3'-dimethoxydiphenyl | Blue. |
| 2,3-hydroxynaphthoyl-aminobenzene | do | 1-amino-4-benzoyl-amino-2,5-dimethoxybenzene | Do. |
| Do | do | 4,4'-diamino-3,3'-dimethoxydiphenyl | Do. |
| Do | C.I. Disperse Blue 79 | 1-amino-4-benzoylamino-2,5-dimethoxybenzene | Do. |
| 1-(2',3'-hydroxynaphthoylamino)-2,4-dimethoxy-5-chlorobenzene | C.I. Disperse Red 17 | 1-amino-2-methyl-5-chlorobenzene | Red. |
| 1-(2',3'-hydroxynaphthoylamino)-2-nethyl-4-chlorobenzene | C.I. Disperse Orange 1 | 1-amino-3-chlorobenzene | Orange. |
| 2,3-hydroxynaphthoyl-aminobenzene | C.I. Disperse Blue 9 | 4,4'-diamino-3,3'-dimethoxydiphenyl | Blue. |

| Coupling component | Disperse dyestuff | Diazotized amine | Shade |
|--|---|--|---------------------|
| 1 - (2',3' - hydroxynaphthoylamino) - 2 - methoxybenzene. | C.I. Disperse Red 17. | 1 - amino - 2 - methoxy - 5 - chloroben- zene. | Red. |
| 1 - (5' - hydroxy - 1',2',1'',2'' - benzocar- bazole - 4' - carboxylamino) - 4 - me- thoxybenzene. | C.I. Disperse Blue 81 plus C.I. Disperse Orange 13 plus C.I. Disperse Red 17. | 1 - amino - 2 - methoxy - 4 - nitroben- zene. | Black. |
| 1 - (2',3' - hydroxynaphthoylamino) - 2 - methylbenzene. | 4-aminobenzaldehydeoxime→1 - (2',3' - hydroxy- naphthoylamino)-4-methoxybenzene. | 1 - amino - 2 - methyl - 5 - chloroben- zene. | Yellowish red. |
| 1 - (2',3' - hydroxynaphthoylamino) - 2 - methoxybenzene. | do. | do. | Do. |
| 1 - (2',3' - hydroxynaphthoylamino) - 2 - methoxybenzene. | 1 - amino - 2 - cyano - 4 - nitraniline → N,N - bis - (β-acetoxyethyl)-aniline. | 1-amino-2-nitro-4-chlorobenzene. 1 - amino - 2 - methoxy - 4 - nitroben- zene. | Red. Bluish red. |
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| 1-(2',3'-hydroxynaphthoylamino)-4- methoxybenzene. | 1-amino-2-cyano-4-nitraniline → N,N-bis-(β-ace- toxyethyl)-aniline plus bromo-1,5-dihydroxy-4, 8-diaminoanthraquinone. | o-Aminoazotoluene. | Scarlet. |
| 1-(2'-hydroxycarbazole-3'-carboxylamino)- 4-chlorobenzene. | 1-amino-4-phenylazonaphthalene → phenol plus 1, 5-dihydroxy-4-hydroxymethylamino-8-aminoan- thraquinone plus 1-amino-2-cyano-4-nitraniline → N,N-bis-(β-acetoxyethyl)-aniline. | 1-amino-2-chloro-5-trifluorobenzene. | Brown. |
| 4,4'-bis-acetoacetyl-amino-3,3'-dimethyl- diphenyl. | Bromo-3'-hydroxyquinophthalone. | 1-amino-2,5-dichlorobenzene. | Yellow. |
| 1-(2',3'-hydroxynaphthoylamino)-4- methoxybenzene. | 1-amino-4-phenylazonaphthalene → phenol plus 1-amino-2-cyano nitraniline → N,N-bis-(β- acetoxyethyl)-aniline plus bromo-1,5-dihydroxy- 4,8-diamino-anthraquinone. | 4-amino-2, 5-dimethoxy-4'-nitro-1, 1'- azobenzene. | Black. |
| 1-(2',3'-hydroxynaphthoylamino)-2- methylbenzene. | Bromo-1,5-dihydroxy-4,8-diaminoanthraquinone. | 4,4'-diamino-3,3'-dimethoxydiphenyl. | Blue. |
| 1-(2',3'-hydroxynaphthoylamino)-2- ethoxybenzene. | do. | do. | Do. |
| 1-(2',3'-hydroxynaphthoylamino)-2- methylbenzene. | do. | 1-amino-4-benzoylamino-2,5-diethoxy- benzene. | Do. |
| 1-(5'-hydroxy-1',2',1'',2''-benzocarbazole- 4'-carboxylamino)-4-methoxybenzene. | Bromo-1,5-dihydroxy-4,8-diaminoanthraquinone plus 1-amino-4-phenylazonaphthalene → phenol plus 1-amino-4-nitrobenzene → 3-methyl-N,N- bis-(β-hydroxyethyl) aniline. | 1-amino-2-methyl-4-nitrobenzene. | Black. |

We claim:

1. A process for dyeing textile materials consisting of mixtures of polyester and cellulose fibers, which comprises treating the textile material with alkaline solutions containing a coupling component, at least one disperse dyestuff, as well as wetting or dispersing agents, drying the material and subsequently treating it with acid solutions which contain, in addition to compounds giving an acid reaction, a diazotized aromatic or heterocyclic amine, drying the material and then subjecting it to a heat treatment.

2. The process as claimed in claim 1, wherein the coupling component is an aromatic or heterocyclic o-hydroxy-carboxylic acid aryl amide or an acylaceto-aryl amide.

3. The process as claimed in claim 1, wherein the

diazo compound is of the benzene, diphenyl or azobenzene series.

4. The process as claimed in claim 1, wherein the disperse dye is of the azo or anthraquinone series.

5. The process as claimed in claim 1, wherein the wetting or dispersing agent is a condensation product of a long chain fatty acid and an aminoalkyl sulfonic acid or of formaldehyde and a naphthalene sulfonic acid or a purified waste lignin liquor.

6. The process as claimed in claim 1, wherein the acid impregnation is effected at a pH in the range of from about 4 to 7.

7. The process as claimed in claim 1, wherein the heat treatment is effected at a temperature in the range of from about 170° to 210° C.

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