

[54] **PROCESS FOR DYEING TEXTILES MADE OF POLYESTER OR CELLULOSE TRIACETATE**

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[51] Int. Cl..... **D06p 1/68**

[58] **Field of Search** ..... 8/170, 174, 94, 175

[56] **References Cited**

**UNITED STATES PATENTS**

2,828,180	3/1958	Sertorio .....	8/62
119,187	9/1871	Simonin .....	8/1 R X
290,110	12/1883	Rau .....	8/1 R

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[57] **ABSTRACT**

Process for dyeing textiles made of polyester or cellulose triacetate with dispersed dyes, in an aqueous medium, whereby the dyeing is carried out with the concurrent use of 0.5 – 10 c.c./l. trichloroethylene and/or perchloroethylene as carrier and an emulsifier, with pressure and temperatures above 100°C..

**7 Claims, No Drawings**

## PROCESS FOR DYEING TEXTILES MADE OF POLYESTER OR CELLULOSE TRIACETATE

It is known to add in dyeing polyester or cellulose triacetate fibers at temperatures of approx. 100°C. special auxiliaries, i.e. so-called carriers to the aqueous dye-bath.

The term "carrier" is to be understood as defining such substances as loosen up the fiber structure and, thus, facilitate the absorption of the dyestuff. The carriers customarily used are aromatic compounds, such as diphenyl, halogeno benzenes, esters of the benzoic, salicylic or cresotic acids, naphthalene and alkyl-naphthalenes, o-phenylphenol and others as well as tripropyl-phosphate. The use of carriers is impaired by a number of disadvantages, such as the formation of unpleasant smells during the dyeing, the toxicity of the carrier and the adverse influence on the fastness to light of the dyeings obtained.

In the last few years one has developed processes according to which textiles are dyed from organic solvents, if required with the addition of water. The organic solvents to be used are alcohols as well as those products as are customarily employed in dry cleaning, such as trichloroethylene and perchloroethylene. In a continuous dyeing and printing process (DOS 2,008,983) an emulsion is used which consists of water and a halogenated aliphatic hydrocarbon, whereby at least one of the phases requires to contain a high-molecular thickening agent. In this process the textile materials are impregnated or printed with the emulsion which contains the dyestuff, the latter being subsequently fixed by steaming or heating. The emulsion contains more than 50 percent by volume, preferably more than 90 percent by volume of the organic phase. The use of large amounts of solvents implies a recovery of the solvents, thus raising the expenses of the dyeing process.

A further known process for dyeing polyester and cellulose triacetate fibers, which is described in DOS 1,918,340, consists in that the fibers are treated with halogenated hydrocarbons prior to getting into contact with the dyeing liquor. This treatment normally consists in that the fibers are placed, for several minutes, into a more or less heated halogenated hydrocarbon solvent. Subsequently, the excess halogenated hydrocarbon solvent is removed by dipping the fiber material into hot water or by treating it with steam. Dyeing is then carried out in an appropriate manner with an aqueous dye-bath being heated to the boil. This process involves, however, the disadvantage that it does require two specific additional pre-treatments, i.e. the placing into a more or less heated halogenated hydrocarbon solvent followed by treatment with hot water or steam. It has been known from DOS 1,918,340, page 3, para 3 that these pretreatments are absolutely necessary for attaining a satisfactory dyeing. Unlike the preliminary impregnation of the fiber with the solvent, the latter addition of the solvent to the dye liquor does not bring about a satisfactory dyeing, because the absorption of the dye is poor and the shades obtained are only weak.

A further process known from French patent 1,141,819 consists in dyeing linear aromatic polyesters by adding to the dye-bath halogenated aliphatic hydrocarbons having a boiling point below 125°C. together with sodium oleyl sulfate as emulsifier. Dyeing is then carried out in an open vessel at temperatures below

100°C. This process leads to only poor dye yields and involves the risk that the dyeings obtained are uneven. Consequently, neither this known process nor the use of halogenated aliphatic hydrocarbons as carriers have been approved of in practice.

It is an object of the present invention to provide a simple and inexpensive process for dyeing textiles made of polyester or cellulose triacetate which avoids the drawbacks inherent in the known processes and pointed out hereinbefore and yields unobjectionable dyeings.

Surprisingly, it has, now, been found that trichloroethylene or perchloroethylene is excellently suited as carriers for dyeing textiles made of polyester or cellulose triacetate provided that the trichloroethylene or the perchloroethylene or a mixture of both is employed in small amounts ranging from 0.5 to 10 c.c./l. and that the dyeing is carried out under pressure at temperatures above 100°C.

Thus, the present invention relates to a process for dyeing textiles made of polyester or cellulose triacetate in an aqueous medium with dispersed dyes, at an elevated temperature and with the use of trichloroethylene and/or perchloroethylene as carrier and an emulsifier. The process of the present invention is characterized in that the dyeing is carried out under pressure at temperatures above 100°C. with 0.5 to 10 c.c./l. of the carrier. Preferably, the carrier is employed in amounts of 1.5 to 5 c.c./l.

The emulsifier to be used according to the present invention may be any known emulsifier, whereby this term is to be understood as defining any substance or mixture of substances preventing the separate phase of an emulsion, in the instant case the individual drops of the trichloroethylene and/or perchloroethylene, from fusing together. One condition of such effect is the activity of the interfaces. Apart from that, a useful emulsifier has to show a stabilizing effect which is due either to the electric charge involving an electrostatic repulsion of the particles, or to the formation of a stable protective film. These effects may also be superposed.

Suitable emulsifiers are for instance: anion active substances, such as alkyl sulfates (for instance sodium lauryl sulfate and sodium cetyl sulfate), Turkey red oils, sulphonated oils, alkyl sulfonates, alkylarylsulfonates, such as alkylbenzyl sulfonates and alkyl naphthalene sulfonates. Further suitable emulsifiers are non-ionic ethyleneoxide and/or propyleneoxide adducts, such as oxethylated alkylphenols, alcohols, aliphatic or unsaturated carboxylic acids, fatty amines, hydroxyl group-containing esters of saturated or unsaturated carboxylic acids. It is also possible to employ mixtures of emulsifiers as are described, for instance, in DOS 1,619,489 and DOS 1,802,210. These mixtures of emulsifiers consist, for instance, of addition compounds obtained by adding ethyleneoxide on alkylphenols (component I), alkali salts of alkylbenzene sulfonic acids (component II) and aliphatic primary or secondary alcohols having three to six carbon atoms (component III).

Particularly good results are obtained if the emulsifier employed is a mixture of oxethylated castor oil and the calcium salt of the dodecylbenzene sulfonic acid, optionally in combination with isobutanol.

In particular, if oxethylated fatty amines are employed, it is advisable to add to the aqueous dye medium a dispersing agent in order to avoid a flocculation

of the standardizing agent which may be present in commercial dispersed dye preparations. Such dispersing agent is, for instance, the sodium methylene-bis-naphthalene sulfonate.

The emulsifier or the mixture of emulsifiers may be added to the aqueous dye liquor. Subsequently, the trichloro or perchloroethylene, or a mixture of trichloro and perchloroethylene may be added with stirring. In general, however, it is simpler and more advisable to add the emulsifier or the mixture of emulsifiers to the trichloro and/or perchloroethylene and to introduce while stirring the mixture thus obtained to the dye liquor.

Related to the carrier, an amount of 5 to 45 percent by weight of emulsifier is employed which means that, if a mixture of the emulsifier and the trichloro or perchloroethylene is used, about 4.8 to 31 percent by weight of emulsifier may be present in such mixture. Normally, it is more advantageous, in particular with a view to the dye yield, to use small amounts of emulsifier i.e. those ranging from 5 to 15 percent by weight related to the carrier, rather than large amounts.

Dyeing is preferably carried out with pressure and at temperatures ranging from 105° to 130°C.

The polyester or cellulose triacetate materials to be dyed may be employed, for instance, in the form of yarn or woven or knitted fabrics. They may furthermore contain other materials, such as wool or polyacrylonitrile.

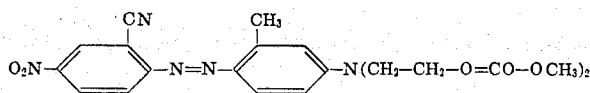
As compared with the known dyeing process, according to which the fabric to be dyed had first to be placed into a halogenated hydrocarbon solvent and then treated with steam or hot water, the process of the present invention is substantially simpler and less expensive and yields dyeings that are at least on a par with the dyeings obtained according to the known process.

The process of the present invention leads in many cases to a substantial improvement of the levelling power of dispersed dyes, which has favorable consequences in particular on the appearance of textured materials. Thus, in particular when dyed onto textured polyester yarn, a barry dyeing may be avoided or at least may thus disadvantage considerably be reduced. Consequently, it is possible to use in the process of the present invention also those dispersed dyes which strongly mark. Such dyes have in most cases excellent fastness properties, but could hitherto practically not be used on textured materials due to the strong marks of the barriness (barre effect). According to the process of the present invention a very good dye yield is obtained which normally exceeds that obtained with the use of the customary carriers. A further advantage of the instant process consists in that the fastness to light of the dyeings obtained is not disadvantageously influenced even if the dyeings are not re-fixed.

The following examples are given for the purpose of illustrating the present invention. The temperatures given are in degrees centigrade and the weights are percent by weight.

#### EXAMPLE 1

A fabric containing polyester fibers that are heat-set in different ways is dyed at a liquor ratio of 1:20 from an aqueous dye-bath with 2.5 percent (related to the weight of the fabric) of a finely dispersed dye of the formula



and with the addition of 3 c.c./l. of a mixture of 95 percent perchloroethylene and 5 percent emulsifier. The temperature is raised within 30 minutes to 120° and dyeing is carried out during 90 minutes at this temperature.

Obtained is an even, settled dyeing, the fastness to light of which is not affected.

Similarly good results are obtained by reducing the dyeing time and/or increasing the dyeing temperature.

The emulsifier employed consists of a mixture of calcium-dodecylbenzene sulfonate and oxethylated castor oil (containing 30 oxethyl groups in the molecule) at a weight ratio of 1:1.

If no perchloroethylene is added, an only unsettled, barry dyeing with a poor yield is obtained.

#### EXAMPLE 2

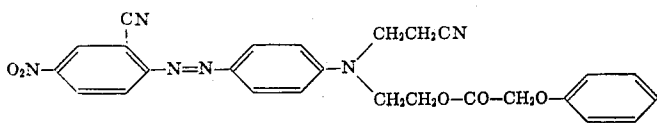
Polyester yarn is dyed at a liquor ratio of 1:10 from an aqueous dye-bath containing 3 percent (related to the weight of the fabric) Palanilviolett 3B (C.I. Disperse Violet 8; C.I. No. 62030), 5 c.c./l. of a mixture of 90 percent perchloroethylene and 10 percent emulsifier as well as 1 g./l. sodium methylene-bis-naphthalenesulfonate. The mixture is heated within 30 minutes to 120° and dyeing is carried out during 90 minutes at 120°. As compared with a dyeing prepared without the concurrent use of perchloroethylene a substantial increase in the depth of the shade is attained without the fastness to light being affected. The emulsifier employed is an ethoxylated coconut oil amine with 10 ethoxyl groups in the molecule as may be prepared by reaction of coconut oil amine with 10 mol ethyleneoxide.

#### EXAMPLE 3

A mixed fabric made of polyester and wool is dyed at a liquor ratio of 1:30 from a dye-bath containing 2 percent (related to the weight of the fabric) of Setacyl-blau PRS (C.I. Disperse Blue 19; C.I. No. 61110), 2 c.c./l. of a mixture of 80 percent perchloroethylene and 20 percent nonylphenoloxethylate with 10 oxethyl groups in the molecule. The temperature is raised in the closed apparatus within 30 minutes to 105° and dyeing is carried out during 90 minutes at this temperature. Obtained is a blue dyeing showing good fastness properties. The dye yield obtained is substantially higher than in the case of the concurrent use of aromatic chlorinated hydrocarbons. A similar result as obtained with perchloroethylene is achieved if instead of perchloroethylene trichloroethylene or a mixture of perchloroethylene and trichloroethylene is employed.

#### EXAMPLE 4

A polyester fabric is dyed in a closed apparatus during 90 minutes at a liquor ratio of 1:20 from a dye-bath containing 3 percent (related to the weight of the fabric) of the finely dispersed dyestuff of the formula



admixed with 3 c.c./l. of a mixture of 95 percent perchloroethylene and 5 percent emulsifier, and 2 g./l. sodium methylene-bis-naphthalenesulfonate, the dye-bath having been heated during 30 minutes to 105°. Obtained is a substantially deeper shade as compared with the dyeings achieved without the addition of perchloroethylene; moreover, the fastness to light is not impaired. The emulsifier employed is a mixture of oxethylated coconut oil amine (with 10 ethoxyl groups in the molecule) and stearylalcohol oxethylate (with 8 ethoxyl groups in the molecule).

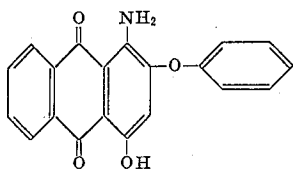
#### EXAMPLE 5

A cellulose triacetate fabric is heated to 105° during 25 minutes in a dye-bath at a liquid ratio of 1:20, the bath containing 2.5 percent (related to the weight of the fabric) Resolinrubin BL (Disperse Violet 40) and 3 c.c./l. of a mixture of 85 percent perchloroethylene and 15 percent emulsifier. Subsequently, it is dyed for 60 minutes at this temperature. As compared with a dyeing without the addition of perchloroethylene one obtains a substantially deeper shade as well as a markedly better appearance of the fabric.

The emulsifier employed is a mixture of oxethylated castor oil (prepared from castor oil and 36 mols ethyleneoxide), calcium dodecylbenzenesulfonate and isobutanol at a weight ratio of 3:2:1.

#### EXAMPLE 6

A polyester fabric is dyed during 30 minutes at 120° and at a liquor ratio of 1:20 from a dye-bath containing 4 percent (related to the weight of the fabric) of a finely dispersed dyestuff of the formula



as well as 2 c.c./l. of a mixture of 5.5 percent emulsifier and 94.5 percent perchloroethylene. After rinsing and working up, one obtains with a good yield an even dyeing.

10 The emulsifier employed is a mixture of oxethylated castor oil (with 36 mols ethyleneoxide), calcium dodecylbenzenesulfonate and isobutanol at a weight ratio of 3:2:1.

15 If a polyester fabric is dyed at temperatures below 100° with the same dyestuff, but analogously to the process described in French patent No. 1,141,819, one obtains dyeings with substantially poorer yields.

According to the instant process it is not only possible to obtain an excellent even appearance of the fabric and a very good dye yield, but also to avoid dye redepositions on the apparatus and on the dyed material. Thus achieved is an improvement of the fastness to rubbing of the dyeing.

We claim:

25 1. A process for dyeing a polyester or cellulose triacetate textile material which comprises dyeing said textile material under pressure and at a temperature above 100°C. with an aqueous dyebath containing dispersed trichloroethylene, perchloroethylene or a mixture thereof, as carrier, in an amount of 0.5-10 c.c./l., an emulsifier and dispersed dye.

2. The process of claim 1 wherein the carrier is present in an amount of 1.5-5 c.c./l.

3. The process of claim 1 wherein said carrier is a mixture of trichloroethylene and perchloroethylene.

35 4. The process of claim 1 wherein said temperature is 105°-130°C.

5. The process of claim 1 wherein said aqueous dyebath contains 5-45 percent by weight, based on the weight of the carrier, of emulsifier.

40 6. The process of claim 1 wherein said aqueous dyebath contains 5-15 percent by weight, based on the weight of the carrier, of emulsifier.

45 7. The process of claim 1 wherein said emulsifier is a mixture of oxethylated castor oil and calcium dodecylbenzene sulfonate.

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