PATENT SPECIFICATION (11) 1 595 984

1 595 984

(21) Application No. 53279/77 (22) Filed 21 Dec. 1977
(31) Convention Application No. 2658062
(32) Filed 22 Dec. 1976 in
(33) Federal Republic of Germany (DE)
(44) Complete Specification published 19 Aug. 1981
(51) INT CL³ D06P 3/82 3/87
(52) Index at acceptance
D1B 2T

(54) PROCESS FOR THE CONTINUOUS DYEING IN ONE BATH OF BLENDED FABRICS OF MODIFIED POLYESTER FIBRES AND WOOL FIBRES

(71) We, HOECHST AKTIENGESELLSCHAFT, a body corporate organised according to the laws of the Federal Republic of Germany, of 6230 Frankfurt/Main 80, Postfach 80 03 20, Federal Republic of Germany, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the dyeing of blended fabrics comprising polyester fibres and wool fibres.

For a long time both one bath and two bath dyeing processes have proved suitable for dyeing blended fabrics consisting of polyester fibres and cellulose fibres. In these processes the polyester fibre component is dyed with disperse dyestuffs according to the so-called thermosoling process, whereby the disperse dyestuffs diffuse under the action of heat into the polyester fibres and are firmly fixed. The original thermosol dyeing process is described in US Patent Specifications Nos. 2 663 612 and 2 663 613.

The dyeing of the cellulose fibre component of these mixtures is carried out either simultaneously with the dyeing of the polyester fibre component or in a separate dyeing operation following or preceding the thermosol dyeing. The one bath dyeing process, in which disperse dyestuffs and the dyestuffs for the cellulose fibres (or other fibre admixtures) are jointly applied to the fabric and then fixed on the fibrous material by the action of hot air, are given special attention, however, owing to the very simple procedure and the fact that the machine has to be loaded only once. References to such processes are to be found in Swiss Patent Specifications Nos. 509 452 and 503 836.

A process known in the art that is of especial interest is a one bath thermosol dyeing process using leuco vat ester dyestuffs, in which both fibre components are simultaneously dyed to the same depth in one process step, namely during the heat treatment. This process is known in expert circles as the ATE process (Anthrasol-Thermosol-Entwicklungsverfahren) and is described in Fischer-Bobsien, Internationales Lexikon Textilveredlung und Genzgebiete (International Dictionary on textile finishing and related fields) 4th Edition (1975), column 104.

Articles in Chemiefaser 13/6 (1963), pages 434—438 and SVF-Fachorgan 16/9 (1961), pages 562—572 examine the thermosoling process for blended fabric consisting of polyester fibres and wool or for wool alone. It is clear in this connection from the latter publication that one bath thermosoling processes are not known for polyester fibre/wool mixtures. Presumably it has not been possible to introduce dyeing methods of this type on account of the totally different behaviour of the wool when dyeing and above all during the thermosoling treatment in comparison with polyester fibres. The prejudice in expert circles against such a process is expressed by the contrast between the small number of literature sources available about this process regarding polyester fibre and wool mixtures and the abundant literature on the thermosol dyeing of polyester/cellulose fibre mixtures.

A possible reason for the supposed impossibility of a joint thermosol dyeing process for polyester fibres and wool is that the hitherto known "normally" dyeable polyester fibres of diethylene glycol and terephthalic acid have to be thermosoled at temperatures above 190°C in order to guarantee full colour yields. This high thermosoling temperature is, however, very detrimental to wool. Among other defects, fibre embrittlement and yellowing occur.

Also known is an exhaust dyeing process in which wool is dyed with leuco vat ester dyestuffs. After absorption of this dyestuff on to the wool, the dyestuff is split by treatment with sulphuric acid/dichromate and oxidised. The known process has scarcely been paid any attention in practice, however, because the oxidation of the leuco compound with dichromate uncontrollably alters the colour shade of the dyestuff produced and furthermore the wool takes on a hard, brittle feel.

There is therefore a need for a process whereby polyester fibre/wool mixtures can be dyed in one bath without damage to the wool by heat and/or the oxidising agent.

We have found that the above disadvantages can be substantially avoided by using in these fibre mixtures instead of "normally" dyeable polyester fibres, polyester fibres that are modified in such a manner that they can be dyed at boiling temperature without the use of carriers.

The present invention provides a process for the continuous or semi-continuous dyeing in one bath of blended fabrics consisting of polyester fibres and of wool fibres with disperse dyestuff and leuco vat ester dyestuff according to a thermosol wet development process, which comprises padding the textile material with an aqueous dye liquor containing the two classes of dyestuff, subjecting to an intermediate drying process and then thermosoling at a temperature in the range of from 160 to 180°C, then passing the textile in a period of 30 to 40 seconds through an aqueous bath at a temperature in the range of from 30° to 40°C containing 5 to 10 ml/l of concentrated sulphuric acid and 0.2 to 0.5 g/l of sodium chlorite, and then passing it through air at room temperature for a period of up to 1 minute, and wherein the polyester fibres used are carrier-free dyeable fibres consisting of polyethylene terephthalate modified with a hydroxy-carboxylic acid and/or an aliphatic dicarboxylic acid and/or of polyethylene terephthalate modified with polyethylene oxide in the form of a block copolymer.

As a result of the combination of a thermosol process with a disperse dyestuff at a low thermosoling temperature (maximum 180°C) and a wet development process for the leuco vat ester dyestuff, the damage to the wool is practically removed. Fast dyeings, which in particular are unexpectedly fast to rubbing, are obtained.

The process of the present invention may advantageously be carried out as follows: The disperse dyestuff is dispersed in a portion of the liquor used for padding heated to 40 to 60°C. The leuco vat ester dyestuff is dissolved in as little boiling water as possible and this solution is then combined with the dispersion of the disperse dyestuff to form the padding liquor. In so far as it is necessary, there may additionally be added to the padding liquor a padding auxiliary, wetting agent or other additive to avoid difficulties that may arise in wetting or dye penetration or to avoid migration of the dyestuff. Padding of the polyester fibre/wool textile is carried out with this padding liquor at 40 to 60°C and at liquor pick-ups of 60 to 100% (of the weight of the dry material). The textile material is then dried at 100 to 120°C suitably for 1 to 3 minutes and subsequently thermosoled for 30 to 60 seconds at 160 to 180°C to fix the disperse dyestuff. In order to develop and fix the leuco vat ester dyestuff, the material so treated is passed through water of a temperature of from 30 to 40°C, which contains concentrated sulphuric acid and sodium chlorite in the above-mentioned concentrations. The duration of this passage through water is 30 to 40 seconds and is followed by passage through air of 1 minute's duration, so that the total acting time of the developing liquor is about 1½ minutes. Subsequently the blended fabric is rinsed with water, if necessary neutralised, and subjected to a customary non-reductive after-treatment. The polyester may be a polyethylene terephthalate/polyethylene oxide block copolymer and/or may be a polyethylene terephthalate modified by a hydroxycarboxylic acid and/or aliphatic dicarboxylic acid or by two or more such acids.

The polyester fibres may be fibres of one or more of these modified polyethylene terephthalates.

The process of the present invention uses one or more disperse dyestuffs and one or more leuco vat ester dyestuffs.

In contrast to the known ATE processes for dyeing polyester/cellulose fibre blended fabrics, in the process of the invention the leuco vat ester dyestuff is not split as a result of the low temperature during thermosoling and is consequently also not oxidised; that is, it cannot dye the polyester fibre constituent under these conditions as is the case with the process mentioned by way of comparison. The leuco vat ester must therefore be developed in a separate developing step that is not damaging to the wool. For this, the development with sulphuric acid and

sodium chlorite was found to be favourable. The original objections to this could be overcome by tests. Sulphuric acid in combination with the oxidising agent neither attacks the wool nor impairs the colour shade of the dyestuff when used in the quantities quoted and in the manner described. The customary development of the leuco vat ester dyestuff with sulphuric acid and sodium nitrite results in a rapid and irreparable yellowing of the wool fibres and is not suitable for carrying out the dyeings according to the invention.

By reducing the thermosoling temperature to 160 to 180°C, the wool is substantially preserved, and there is no substantial damage caused.

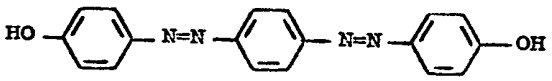
A surprising factor in this continuous process is the exceedingly good fixing of the leuco vet ester dyestuff on the wool. It could not be expected that the dyestuff would be practically completely absorbed on to the wool in the short time available between the padding operation and drying. On the contrary it had been expected that the dyestuff, which is present as a colour pigment after splitting and oxidation, would come away from the fibre and be rinsed off in the after-treatment. At least acid dyestuffs, and undeveloped water-soluble leuco vat ester dyestuffs are chiefly to be regarded as such, cannot be fixed on wool in such a short time.

The following Examples illustrate the invention.

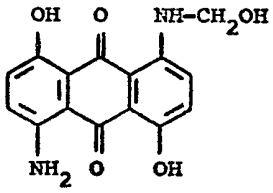
Example 1

A padding liquor was prepared with water of a temperature of 60°C and contained per litre:

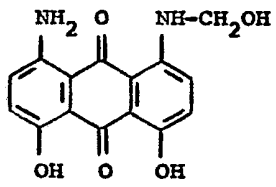
17 g of the yellow disperse dyestuff of the formula



5 g of a blue disperse dyestuff consisting of a mixture (in equal proportions) of the dyestuffs of the formulae

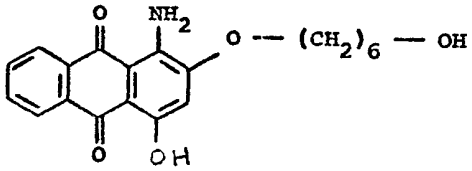


and



and

1.5 g of the red disperse dyestuff of the formula



20 g of the leuco vat ester dyestuff Solubilised Vat Green 3, Colour Index No. 69 501

(the dyestuffs are in the commercial composition and form) and

5 g of the sodium salt of an alkanesulphonic acid having 12 to 17 carbon atoms in the alkyl radical

3 g of the reaction product of 1 mole of isotridecyl alcohol with 8 moles of ethylene oxide and

2.5 g of triisobutyl phosphate.

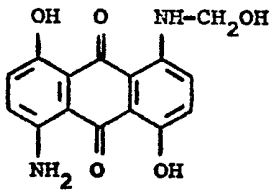
A gabardine blended fabric consisting of 55% fibres of a polyethylene terephthalate/polyethylene oxide block copolymer and 45% wool was padded with this liquor at a liquor pick-up of 70% (of the weight of the dry material).

After padding, the textile material was dried at 100°C and then thermosoled for 60 seconds at 175°C to fix the disperse dyestuff. Passing the material for 30 seconds through an aqueous bath of a temperature of 30°C containing

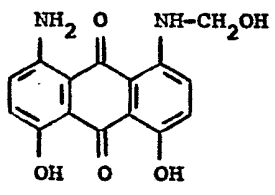
10 cm^3/l of concentrated sulphuric acid and
0.5 g/l of industrial grade sodium chlorite (50% strength)
followed by passing through air for 1 minute at room temperature caused the leuco vat ester dyestuff on the wool to develop. Subsequently the dyeing was rinsed with water and after-treated for 10 minutes at 70°C in an aqueous bath containing
2 cm^3/l of acetic acid (60% strength) and
5 g/l of an auxiliary mixture comprising
50% by weight of the reaction product of 1 mole of castor oil with 36 moles of ethylene oxide,
35% by weight of the calcium salt of tetrapropylenesulphonic acid and
15% by weight of isobutanol.
The dyed blended fabric was finally rinsed again with warm water (40°C) and cold water.
An olive-coloured fabric of good solid shade, which consisted of the polyester/wool mixture, was obtained.

Example 2

The dyeing procedure was the same as in Example 1, except that in this case the following dyestuffs were used in the quantities given below:
20 g/l of a blue disperse dyestuff consisting of a mixture (in equal proportions) of the dyestuffs of the formulae



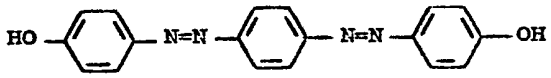
and



and
15 g/l of the leuco vat ester dyestuff Solubilised Vat Blue 6, Colour Index No. 69 826.
After intermediate drying, the material was in this case thermosoled for 60 seconds at 160°C; otherwise the procedure for the development of the leuco ester to form the vat dyestuff and for the after-treatment of the dyeing was exactly the same as that in Example 1.
A clear blue dyeing of the polyester/wool fabric with a good solid shade for both fibre constituents as well as very good fastness to rubbing was obtained.

Example 3

The procedure as in Example 1 was employed, except that the following dyestuffs in the amounts indicated below were used:
30 g/l of the yellow disperse dyestuff of the formula



and
20 g/l of the leuco vat ester dyestuff Solubilised Vat Orange 1, Colour Index No. 59 106.
In other respects the dyeing process was exactly the same.
A strong gold-yellow coloured material comprising the fibrous mixture was obtained with a very good solid covering of the two fibre constituents and with good fastness to rubbing.

WHAT WE CLAIM IS:—

1. A process for the continuous or semi-continuous dyeing in one bath of blended fabrics consisting of polyester fibres and wool fibres with one or more disperse and one or more leuco vat ester dyestuffs according to a thermosol wet development process, wherein the textile material is padded with an aqueous dye liquor containing the dyestuffs of the two classes, after intermediate drying is thermosoled at a temperature in the range of from 160 to 180°C, then passed in a

period of from 30 to 40 seconds through an aqueous bath at a temperature of from 30 to 40°C containing 5 to 10 ml/l of concentrated sulphuric acid and 0.2 to 0.5 g/l of sodium chlorite, then passed through air at room temperature for a period of up to 1 minute, and wherein the polyester fibres are carrier-free dyeable fibres consisting of polyethylene terephthalate modified with a hydroxycarboxylic acid and/or an aliphatic dicarboxylic acid, and/or polyethylene terephthalate modified with polyethylene oxide in the form of a block copolymer.

2. A process as claimed in claim 1, wherein the intermediate drying is carried out at a temperature in the range of from 100 to 120°C.

3. A process as claimed in claim 1 or claim 2, wherein the thermosoling is carried out for 30 to 60 seconds.

4. A process as claimed in any one of claims 1 to 3, wherein the padding is carried out at a temperature in the range of from 40 to 60°C.

5. A process as claimed in claim 1, carried out substantially as described herein.

6. A process as claimed in claim 1, carried out substantially as described in any one of the Examples 1 to 3 herein.

7. A blended fabric consisting of modified polyester fibres as specified in claim 1 and wool fibres which have been dyed by a process as claimed in any one of claims 1 to 6.

ABEL & IMRAY,
Chartered Patent Agents,
Northumberland House,
303—306 High Holborn,
London, WC1V 7LH.

Printed for Her Majesty's Stationery Office, by the Courier Press, Leamington Spa, 1981
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.