METHOD FOR THERMOFIXATION DYEING OF POLYESTER-COTTON FABRIC IN A MIXTURE OF VAT/DISPERSE DYE

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ABSTRACT OF THE DISCLOSURE

Method for dyeing of fabric, such as polyester/cotton fabric, in a vat/disperse dye system, using hydroxylamine sulfate under conditions of thermal fixation. The hydroxylamine sulfate is maintained at a pH in the range 5.0 to 6.5 in a vat/disperse dye pad to obtain maximum penetration of the dye within the fibers of the fabric.

BACKGROUND OF THE INVENTION

(1) Field of the invention

Continuous dyeing of fabrics made from polyester/cotton blends has been accomplished, as early as 1952 by a plurality of dyeing systems, designated as "resin bonded pigment," "resin bonded pigment-disperse," "sulphur-disperse," "azoic-disperse," "fiber reactive" and "vat dyestuff." Increasing attention to the technique of obtaining optimum penetration of the dye into the fibers is the result of the rapidly mushrooming growth of polyester/cotton blends. In most dyeing systems the dye range equipment includes a dye padded, an infrared drying unit, controlled heat drying (such as steam cans or hot air or both), a high temperature thermofixation unit, a chemical pad, a steamer, a plurality of wash boxes for washing, oxidizing, washing, soaping and washing, and finally a drying system of steam cans or a hot air oven.

(2) Description of the prior art

In the prior art, the vat/disperse combination dye accounts for probably the largest poundage being applied to polyester/cotton today. Some are sold as a mixture by dyestuff companies or put together by the dyer in his own formulation. The pad bath containing the vat/disperse dye has an anti-migrant and is run at 70–80°F. Infrared, controlled drying and thermofixation for 60 to 90 seconds at 400–415°F. are followed by reduction with regular chemical pad (80–100°F.) using caustic soda and sodium hydrosulfite, followed by steaming for 25 to 40 seconds at 212°F. and oxidizing and soaping aftertreatment.

The strong points of such a conventional vat/disperse system are: (a) generally excellent all-round fastness; (b) wide range of dyestuffs for both cotton and polyester; (c) dyeing of heavy shades feasible; and (d) capability of achieving cross-dye effects by choosing proper dyestuffs.

Limitations of the vat/disperse system are: (a) some of the brighter shades must be switched to azoic or fiber reactive dyeing systems; (b) disperse colors must be carefully chosen for thermofixation fastness; and (c) crocking in heavy shades can be excessive.

SUMMARY OF THE INVENTION

The present method distinguishes from the prior art in its capability of enhancing the dye penetration of the fibers. Applicant utilizes hydroxylamine sulfate solution together with the vat/disperse dye as a dyeing auxiliary. By maintaining the hydroxylamine sulfate at the maximum benefit pH range 5.0 to 6.5, the hydroxylamine sulfate dyeing auxiliary enhances penetration of both vat and disperse dye particles into polyester and cotton fiber during thermofixation and, thereby, contributes materially to the finally achieved shade of the fabric. As a result, there is provided a considerable color yield increase and improved shade leveling. Since there is increased dye penetration in the fibers, less dyestuffs are required and more level dyeings are obtainable. A 50/50 blend of hydroxylamine sulfate and anhydrous sodium acetate is preferably used in solution with the vat/disperse dye.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a flow sheet depicting thermofixation dyeing of fabric, according to the present invention and using a suggested range of dyeing equipment.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Laboratory dyeings were carried out with polyester/cotton fabric (50/50 blend) using the vat/disperse dye system and the pad-thermofixation dyeing method to determine what benefits might be derived from the use of hydroxylamine sulfate as a dyeing auxiliary.

The pad-thermofixation dyeing procedure is the most widely used method for the continuous dyeing of polyester/cotton fabric with vat/disperse dyes. The following dyeing range was set up as pad-thermofixation facility, prepare dye pad (300 mL)

x oz./gal. disperse dye
x oz./gal. vat dye
Pad fabric at 160°F. for 2 minutes
Nip roll squeeze (60% wet pick-up)
Attach fabric to tender frame form
Pre-dry in oven for 30 seconds at 250°F.
Bone dry in second oven at 220°F.
Cure at 410°F. for 90 seconds
Reduction bath:
8 oz./gal. caustic
8 oz./gal. sodium hydrosulfite
Immerse for 5 minutes at 120°F.
Rinse
Oxidize with one oz./gal. sodium perborate solution at 120°F. for five minutes
Soaip with one oz./gal. Naconal NA (Mfg. by National Aniline) at 180°F.
Rinse
Dry

PART I

Initial tests

In the pad-thermofixation dyeing of polyester/cotton fabrics, the fabric is passed through a dye pad containing the vat and disperse dyes. It is then dried and passed through pre-dye and thermofixation units where the heat drives the disperse dye into the polyester fiber by solid state dyeing. The vat dyes present on the surface of the fabric also stain the polyester fiber and add to the buildup of the shade. The migration of the vat dye particles into the polyester fiber during the thermofixation treatment is important to the final shade of the fabric.

Initial dyeings were made with .25 oz./gal. hydroxylamine sulfate of 75% assay using 2.0 oz./gal. shades of Vat Blue 6 and Vat Brown. The hydroxylamine sulfate was added to the dye pad containing the vat dye. Control dyeings without hydroxylation sulfate were also made for shade comparison.

Additional dyeings were made in which Glauber Salt and sulfuric acid were used as auxiliaries in the dye pad to determine if these chemicals alone, which are present in the hydroxylamine sulfate product, were beneficial.
3 Results

(1) Hydroxylamine sulfate, when used in the pad dye-bath with vat dyes, resulted in a considerable increase in color yield and improved shade leveling.

(2) Glauber Salt and sulfuric acid when used in the vat dye bath alone did not exhibit these shade benefits.

PART II

100% cotton dyeings

Dyeings on 100% cotton fabric using Vat Blue 6 and the pad-thermo-fixation method were made to determine if the increase in the color yield was occurring on the cotton or on the polyester portion of the blend. These dyeings indicated that the increase in the color yield resulted from vat dye penetration to both fibers of the blend fabric.

Extraction tests were carried out on polyester/cotton dyeings with vat dyes where the cotton portion of the blend was leached out with sulfuric acid (70%), leaving the dyed polyester fiber to evaluate for shade. These tests showed increased color value of vat dye on the polyester fiber when hydroxylamine sulfate was present in the pad bath.

PART III

Optimum conditions

A series of dyeings were made with 0.05, 0.10, 0.15, and 0.20 ounce per gallon hydroxylamine sulfate of 98% assay concentrate using 2 ounces/gallon shades of Vat Blue 6 on polyester/cotton fabric to determine the concentration necessary to obtain maximum shade benefit. The dyeing where 0.10 ounce/gallon hydroxylamine sulfate (98% assay) was used in the dye pad resulted in the best color yield.

An additional dyeing series was made with 0.10 ounce/gallon hydroxylamine sulfate (98% assay) and 2 ounces/gallon Vat Blue 6 to determine the pH range for maximum color value. The pH's of the dyeings were adjusted to 5.0, 5.5, 6.0, 6.5 with 0.5 N caustic. The results of these tests indicated that maximum benefit was obtained in the pH 5.0–5.5 range.

Results

The above dyeings indicated that maximum color yield was obtained when 0.10 oz./gal. hydroxylamine sulfate (98% assay) was present in the dye pad bath adjusted to pH 5.5.

PART IV

Hydroxylamine mixtures

Observations made from previous dyeings indicated that a compound with alkaline properties needed to be present in an hydroxylamine sulfate mixture to neutralize the sulfuric acid present in hydroxylamine sulfate and also to assist in increasing the pH of the dyebath to the optimum 5.0–5.5 level.

Mixtures of hydroxylamine sulfate with alkaline compounds such as sodium bicarbonate, sodium acetate, sodium tartrate, and sodium borate were tried in the dye pad bath. It was found that a 50/50 mixture of hydroxylamine sulfate and amphoteric sodium acetate gave the best dyeing results of any of the compounds tried. In fact, better color yield results were obtained with this mixture than on any of the dyeings made to this point.

Shelf life of the 50/50 hydroxylamine sulfate-sodium acetate mixture was checked over a four-week period for stability. The hydroxylamine sulfate assay after four weeks remained at 50% and the blend mixture was free-flowing with no apparent caking present.

An attempt was made to get away from the acid nature of hydroxylamine sulfate by making a small amount of hydroxylamine oxalate in the laboratory and carrying out polyester/cotton dyeings to determine its effectiveness. These dyeings with hydroxylamine oxalate and Vat Blue 6 did not exhibit as much color value increase as obtained in prior hydroxylamine sulfate or hydroxylamine sulfate-sodium acetate dyeings.

PART V

Vat/disperse dyeings with 50/50 hydroxylamine sulfate-sodium acetate mixture

Dyeings were made with vat/disperse dyes and the hydroxylamine sulfate-sodium acetate mixture on 50/50 polyester/cotton and 65/35 polyester/cotton fabric using the pad-thermo-fixation method. Four different shades were dyed representing actual plant dyehouse shades for uniform wear.

The hydroxylamine sulfate-sodium acetate mixture was dissolved in a small amount of water before addition to each of the dye pad baths. The application concentration of the mixture was from 0.5–1.0 oz./gal., depending on the depth of shade. The dye formulas for these shades were:

**DYE FORMULAS 1 FOR DYEING OF POLYESTER/COTTON FABRIC WITH VAT/DISPERSE DYES AND HYDROXYLAMINE SULFATE BY THE PAD-THERMOFIXATION METHOD**

<table>
<thead>
<tr>
<th>Blue shade</th>
<th>Oz./gal.</th>
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<tr>
<td>Calcosperse Navy Blue RK</td>
<td>0.55</td>
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<tr>
<td>Calcosperse Red GF</td>
<td>0.19</td>
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<td>Calcosperse Black FG</td>
<td>0.40</td>
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<tr>
<td>Calcoloid Blue BLRC</td>
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<tr>
<td>Calcoloid Violet BNC</td>
<td>1.89</td>
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<tr>
<td>Calcoloid Olive Green BDC</td>
<td>0.90</td>
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<table>
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<tbody>
<tr>
<td>Calcosperse Yellow RL</td>
<td>1.10</td>
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<tr>
<td>Calcosperse Orange RLK</td>
<td>0.66</td>
</tr>
<tr>
<td>Calcosperse Navy RK</td>
<td>1.80</td>
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<tr>
<td>Calcoloid Direct Black PBC</td>
<td>2.40</td>
</tr>
<tr>
<td>Calcoloid Olive Green BDC</td>
<td>3.20</td>
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<tr>
<td>Calcoloid Jade Green NC</td>
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<table>
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<td>Calcosperse Black FG</td>
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<td>Calcosperse Navy Blue RK</td>
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<tr>
<td>Calcosperse Red GF</td>
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<tr>
<td>Calcoloid Blue BLRC</td>
<td>3.8</td>
</tr>
<tr>
<td>Calcoloid Violet BNC</td>
<td>3.5</td>
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<tr>
<td>Calcoloid Direct Black PBC</td>
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<th>Charcoal shade</th>
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<tr>
<td>Calcosperse Black KD</td>
<td>4.10</td>
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<tr>
<td>Calcosperse Orange RLK</td>
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<tr>
<td>Calcoloid Direct Black PBC</td>
<td>3.60</td>
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<tr>
<td>Calcoloid Dark Brown VRF</td>
<td>1.20</td>
</tr>
<tr>
<td>Calcoloid Olive Green BDC</td>
<td>0.80</td>
</tr>
</tbody>
</table>

1 All dyes obtained from American Cyanamid Co.

Results

(1) All dyeings where the 50/50 hydroxylamine sulfate-sodium acetate mixture was used in the vat/disperse dye pad bath showed a considerable increase in color yield when compared to the control dyeing.

(2) It was found that the amount of hydroxylamine sulfate-sodium acetate mixture required to obtain maximum color yield was dependent on depth of shade. Heavy shades (24 oz./gal. total vat/disperse dye) required as much as 1.0 oz./gal. of mixture.

**SUMMARY OF RESULTS**

(1) Hydroxylamine sulfate, when added to the vat/disperse dye pad bath in the pad-thermo-fixation method of dyeing polyester/cotton blend fabric, results in a considerable color yield increase and improved shade leveling.

(2) Shade improvement in color yield and leveling is due to increased staining or dyeing of the cotton and polyester fiber portions of the blend by the vat and disperse
dyes resulting from the presence of hydroxylamine sulfate.

(3) A 50/50 blend product of hydroxylamine sulfate
Conc. and anhydrous sodium acetate was found to be most
effective in this application.

Manifestly, various types of soluble alkali metal salts
may be employed together with the hydroxylamine sul-
fate and the proportion of hydroxylamine sulfate to caustic
and vat/disperse dyestuff may be varied without de-
parting from the spirit and scope of invention.

What is claimed is:

1. Method for thermofixation dyeing of polyester/cot-
ton fabric comprising:
(A) immersing said fabric with a mixture of:
(i) vat/disperse dye; and
(ii) hydroxylamine sulfate (0.05-1.0 ounce per
gallon);
(B) drying said fabric and thermofixation in the ap-
proximate range 400-415°F;
(C) reducing fabric impregnated with dye in caustic-
sodium hydrosulfite solution;
(D) rinsing said fabric with soap and water; and
(E) final drying.

2. Method for thermofixation dyeing of polyester/cot-
ton fabric comprising:
(A) immersing said fabric with a mixture of:
(i) vat/disperse dye; and
(ii) hydroxylamine sulfate maintained by buffer
salts at a pH in the range 5.0-6.5;
(B) drying said fabric by squeezing and heating and
thermofixation in the approximate range 400-415°F;
(C) reducing fabric impregnated with dye in caustic-
sodium hydrosulfite solution;
(D) rinsing said fabric;
(E) oxidizing said fabric with sodium perborate; and
(F) soaping said fabric, rinsing and final drying.

3. Method for thermofixation dyeing of polyester/cot-
ton fabric in a vat/disperse dye system comprising:
(A) preparing a dye pad with a mixture of:
(i) vat/disperse dye; and
(ii) a 50/50 mixture of hydroxylamine sulfate and
sodium acetate (0.5-1.0 ounce per gallon);
(B) advancing said fabric through said pad at a tem-
perature of 160°F;
(C) squeezing said fabric to pick up a major portion
of said soaking mixture;
(D) predrying said fabric for thirty seconds at 250°F;
(E) curing said fabric at 410°F for 90 seconds;
(F) reducing fabric impregnated with dye in a mixture
of caustic (8 ounces per gallon) and sodium hydro-
sulfite (8 ounces per gallon) for five minutes at 120°F;
(G) rinsing said fabric;
(H) oxidizing said fabric in a solution of sodium per-
borate (1 ounce per gallon) at 120°F for five
minutes;
(I) soaping said fabric at 180°F;
(J) rinsing said fabric; and
(K) drying said fabric.

4. Method for thermofixation dyeing of polyester/cot-
ton fabric as in claim 3, including the amount of the
hydroxylamine sulfate-sodium acetate mixture de-
pending on the depth of dye shade desired.

5. Method for thermofixation dyeing of polyester/cot-
ton fabric as in claim 4, including maintaining of said so-
lution of hydroxylamine sulfate and sodium acetate at a pH
in the range 5.0-6.5.

6. Method for thermofixation dyeing of polyester/cot-
ton fabric as in claim 3, wherein the immersing mixture
consists of:
(i) vat/disperse dye; and
(ii) a 50/50 mixture of hydroxylamine sulfate and
sodium bicarbonate (0.5 to 1.0 ounce per gallon).

7. Method for thermofixation dyeing of polyester/cot-
ton fabric as in claim 3, wherein the immersing mixture
consists of:
(i) vat/disperse dye; and
(ii) 50/50 mixture of hydroxylamine sulfate and sodi-
num borate (0.5 to 1.0 ounce per gallon).

9. Method for thermofixation dyeing of polyester/cot-
ton fabric in a vat/disperse dye system comprising:
(A) preparing a dye pad with a mixture of:
(i) vat/disperse dye; and
(ii) a 50/50 mixture of hydroxylamine sulfate
(98% assay) (0.10 ounce per gallon) adjusted
to pH 5.5 with caustic;
(B) advancing said fabric through said pad at a tem-
perature of 160°F;
(C) squeezing said fabric to pick up a major portion
of said soaking mixture;
(D) predrying said fabric for thirty seconds at 250°F;
(E) curing said fabric at 410°F for 90 seconds;
(F) reducing fabric impregnated with dye in a mixture
of caustic (8 ounces per gallon) and sodium hydro-
sulfite (8 ounces per gallon) for five minutes at 120°F;
(G) rinsing said fabric;
(H) oxidizing said fabric in a solution of sodium per-
borate (1 ounce per gallon) at 120°F for five
minutes;
(I) soaping said fabric at 180°F;
(J) rinsing said fabric; and
(K) drying said fabric.

10. Method for thermofixation dyeing of polyester/cot-
ton fabric comprising:
(A) immersing said fabric with a mixture of:
(i) vat/disperse dye; and
(ii) hydroxylamine oxalate maintained by soluble
alkali metal salts at a pH in the range 5.0-6.5;
(B) drying said fabric by squeezing and heating and
thermofixation in the approximate range 400-415°F;
(C) reducing fabric impregnated with dye in caustic-
sodium hydrosulfite;
(D) rinsing said fabric;
(E) oxidizing said fabric with sodium perborate; and
(F) soaping said fabric, rinsing and final drying.

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