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3,504,996

DYEING OF POLYESTER AND CELLULOSE FIBER BLENDS WITH A SILICONE-CONTAINING SOLUTION OF A REACTIVE DYE AND A DISPERSE DYE AND AN AMINOPLAST PRECURSOR TREATMENT THEREOF

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4 Claims

ABSTRACT OF THE DISCLOSURE

A process for the simultaneous dyeing, water-proofing and resin treatment of fabrics containing mixtures of polyester and/or cellulose tri-acetate fibers, cellulosic fibers, which react with cellulose-reactive dyestuffs and the aforesaid resin-forming composition, said process comprising treating said fabrics in an aqueous medium comprising at least a silicone water-proofing composition, an alkaline catalyst, a dyestuff which dyes each of the fibers in the fabric, and stiffening resin, followed by drying the treated fabric and then heat treating the fabric at a temperature of at least 100° C.

This application is a continuation-in-part of Ser. No. 362,173, now abandoned.

The present invention relates to finishing treatments of fabrics of mixtures of cellulosic and polyester fibers and/or cellulose tri-acetate.

It is an object of the present invention to provide a single-bath process for treating such fabrics to dye them and to give them a water-repellent finish simultaneously.

A process for the combined dyeing and finishing of cellulosic fabrics has been described in The Journal of the Society of Dyers and Colourists, vol. 78 (February 1962) at pp. 69-76 and in the specification of United Kingdom Patent No. 846,505. This process employs a cellulose-reactive dyestuff, a resin-forming composition and an acid catalyst in an aqueous medium to confer wash fast crease resisting properties and coloration to the fabrics.

We now provide a process whereby water-repellent properties are imparted in the same bath simultaneously with the coloration treatment and the application of the aforesaid resin-forming composition.

For the coloration to be effective, reactive dyes are used for the natural cellulosic fibers such as cotton or the regenerated cellulosic fibers such as viscose rayon.

Disperse dyes are used for the coloration of polyester fibers and/or cellulose tri-acetate fibers.

The stiffening or crease-resisting resin is included to inhibit swelling of the cellulosic fibers other than cellulose tri-acetate, such as cotton or regenerated cellulose fibers such as viscose rayon.

The polyester fiber may be of homopolymer such as polyethylene terephthalate or a copolymer such as polyethylene terephthalate with a minor proportion of polyethylene isophthalate.

Suitable silicone water-proofing compositions for use with the process are those known to impart water-repellency to textile materials, such as, for example, an emulsion of methyl hydrogen polysiloxane. The inorganic bases sodium sesquicarbonate and sodium bicarbonate,

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are alkaline catalysts which may be used, avoiding bath instability. Sodium bicarbonate is preferred as it gives good results.

By a "disperse dyestuff" we mean a dyestuff which is applied as a suspension with a dispersing agent and fixed to the fiber by the application of heat. The dyestuffs sold under the registered trademarks "Dispersol" and "Duranol" are suitable for use in the process of the invention.

The heat treatment is carried out at a temperature which is at the same time high enough to bond the disperse dyestuff and to cure the silicone water-proofing composition. The preferred range of curing temperatures is 110-220° C., and that of curing times 15 seconds to 5 minutes.

The amount of silicone composition on the finished fabric is preferably from 0.5 to 5% by weight of the fabric.

A shade change normally occurs with disperse dyestuffs on the application of silicone water-proofing compositions. This is largely overcome when using the process of the present invention since the dyer matches to the silicone composition finished shade.

The process of the invention gives fast-dyed, water-repellent fabrics which are particularly suitable for rainwear.

Suitable cellulose-reactive stiffening or crease-resisting resins include melamine formaldehyde, dimethylol-ethylene urea, triazone and methylated urea formaldehyde resins. The resins should, in the presence of a suitable alkaline catalyst, prevent swelling without inhibiting water-proofing with the silicone compositions and impart crease-resisting properties to the cellulosic fibers, other than cellulose tri-acetate.

Suitable cellulose-reactive dyestuffs are those in which the reactive component is a cyanuryl chloride; i.e. these dyes are derivatives of 1:3:5 triazine which contain reactive chlorine atoms attached to the carbon in the triazinyl ring. They are described, e.g. in the paper "The Reactions of Cold-dyeing Procion Dyes With Cellulose" by T. L. Dawson, A. S. Fern and C. Preston published in the Journal of the Society of Dyers and Colourists, vol. 76 (1960) from p. 210 onwards. Reference also may be made to the description of these dyes in British Patents 797,946 and 798,121, viz. monoazo or polyazo dyes (797,946) or anthraquinone dyes (798,121) "... containing a primary or secondary amino-group carrying as N-substituent a 1:3:5 triazine radical containing at least one halogen atom attached to a carbon atom of the triazine ring."

Typical dyes of this type are:

Procion Brilliant Blue R (Chemical Abstracts, p. 883s, July to December subject Index of 1967, C.I. Reactive Blue 4) (now—Brilliant Blue M-R)
Procion Brilliant Red 2B (C.I. Reactive Red 1, a dichlorotriazinyl-monoazo dye) (now—Brilliant Red M-2B)
Procion Brilliant Red 5B (C.I. Reactive Red 2, a dichlorotriazinyl monoazo dye) (now—Brilliant Red M-5B)
Procion Brilliant Red H-3B (C.I. Reactive Red 3, a monochlorotriazinyl-monoazo dye)
Procion Blue H-B (C.I. Reactive Blue 2, a monochlorotriazinyl anthraquinone dye)
Procion Blue 3G (C.I. Reactive Blue 1, a di-chlorotriazinyl anthraquinone dye) (now—Blue M-3G)
Procion Red G (C.I. Reactive Red 5, a di-chlorotriazinyl monoazo dye) (now—Red M-G)
Procion Red 2B (di-chlorotriazinyl monoazo dye, C.I. Reactive Red 1) (now—Red M-2B)
Procion Yellow R (C.I. Reactive Yellow 4, di-chlorotriazinyl monoazo dye) (now—Yellow M-R)

Preferred embodiments of the present invention will now be described hereinafter by way of example.

EXAMPLE 1

A padding bath was prepared as follows:

50 parts of 60% methyl hydrogen polysiloxane emulsion was diluted with a small quantity of water and to this was added 50 parts of methylated urea-formaldehyde resin mixed with a further small quantity of water. 50 parts urea, and 10 parts of sodium bicarbonate were then dissolved in water and added to the bath. 5 parts of "Procion" Brilliant Yellow 6GS (Colour Index Reactive Yellow 1) was dissolved in water and added to a dispersion of 6.5 parts of "Dispersol" (Colour Index Disperse Yellow 1 (C.I. 10345)) Yellow A paste in water, the volume of dyestuff liquid being made up to 70 parts of water, and the dispersion being allowed to stand for ten minutes with occasional stirring. The dyestuff was then strained through a 240 B.S.S. mesh into the padding bath, which was then made up to 1,000 parts with water.

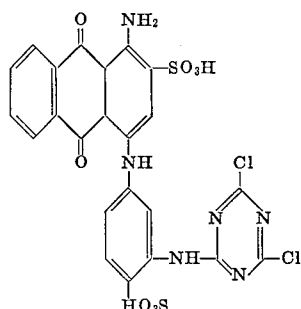
A fabric of 67/33 polyethylene terephthalate/cotton poplin was immersed in the so-prepared padding bath and squeezed through a mangle at a 65% expression. The treated fabric was dried at 120° C. and then cured at 200° C. for 90 seconds. Finally, the fabric was washed with 0.2% soap solution at 45° C., rinsed thoroughly with water until a clear rinse resulted, dried and left for 24 hours before testing.

The resulting fabric has a pleasant handle, the dyes were fast to washing and rubbing and had a spray rating of 100 (ASTM D583-58).

It also proved acceptable when subjected to the Bundesmann tests described in the British Standards Handbook No. 11 (1956). To be acceptable, the absorption was required to be less than 20 initially, less than 30 after five cycles of hand washing, and less than 30 after five cycles of dry-cleaning. Penetration was required to be less than 10 then 15 after 5 cycles D.C., 5 cycles H.W.

EXAMPLE 2

A padding bath was prepared by diluting 45 parts of methyl hydrogen polysiloxane emulsion with a small quantity of water and adding to this 50 parts of methylated urea-formaldehyde resin with a further small quantity of water. A solution of 9 parts of sodium bicarbonate and 50 parts of urea in water was added to the bath, and lastly a solution of 5 parts of "Procion" Brilliant Blue RS (Colour Index Reactive Blue 4 (Chemical Abstracts Subject Index, p. 883s, and British Patent 798,121)) which has the formula:



in water was strained into the bath through a 240 B.S.S. mesh sieve and the bath made up to 1,000 parts with water.

A 100% cotton poplin fabric was padded through the padding bath so prepared and mangled to a 75% expression. After drying at 120° C. the treated cotton fabric was cured for five minutes at 180° C. before scouring in 0.2% soap solution at 45° C. and finally rinsed in water to give a clear rinse. It was then dried and left for 24 hours before testing.

The dyeing of the fabric was wash and rub-fast. The treated fabric had a spray rating of 100 and proved acceptable when subjected to the Bundesmann tests,

EXAMPLE 3

A padding bath was prepared as follows:

50 parts of methyl hydrogen polysiloxane emulsion was diluted with a small quantity of water and to this was added 50 parts of methylated urea formaldehyde resin with a further small quantity of water. A solution of 10 parts of sodium bicarbonate and 50 parts of urea in water was added to the bath 5 parts of "Duranol" Blue G paste (Colour Index Disperse Blue 26 (C.I. 63305)) and 3 parts of "Procion" Brilliant Blue RS (Colour Index Reactive Blue 4) in water were sieved into the bath through a 240 mesh B.S.S. sieve and the bath made up to 1000 parts with water.

A 67/33 polyethylene terephthalate/linen rainwear fabric was padded through the padding bath and squeezed to retain 65% of the liquor. After drying at 120° C. the treated fabric was aired at 200° C. for 90 seconds. The resulting aired fabric was scoured in 0.2% soap solution at 45° C. for ½ hour and rinsed with water until a clear rinse resulted. It was then dried and left for 24 hours before testing.

The dyed fabric was fast to washing and rubbing and had a spray rating of 100. It was also acceptable to the Bundesmann test as described in Example 1.

EXAMPLE 4

A padding bath was prepared as follows:

50 parts of methyl hydrogen polysiloxane emulsion was diluted with a small quantity of water and to this was added a solution of 10 parts of sodium bicarbonate, and 50 parts of urea in water. 5 parts of "Duranol" Blue G paste (Colour Index Disperse Blue 26) was diluted with 65 parts of water, allowed to stand for ten minutes with occasional stirring, and then strained through a 240 B.S.S. mesh sieve into the padding bath which was then made up to 1,000 parts with water.

A fabric comprising 100% cellulose triacetate fibers was immersed in the so-prepared padding bath and squeezed through a mangle at a 65% expression. The treated fabric was dried at 120° C. and then cured at 200° C. for 90 seconds. Finally, the fabric was washed with 0.2% soap solution at 45° C., rinsed thoroughly with water until a clear rinse resulted, dried and left for 24 hours before testing.

The resulting dyed fabric was fast to washing and to wet and dry rubbing, and had a spray rating of 100 (ASTM D583-58). It also proved acceptable when subjected to the Bundesmann tests described in the British Standards Handbook No. 11 (1956). To be acceptable, the absorption was required to be less than 20 initially, less than 30 after five cycles of hand washing and less than 30 after five cycles of dry-cleaning. Penetration was required to be less than 10 initially, less than 15 after five cycles of hand washing and less than 15 after five cycles of dry-cleaning. In fact, the penetration values obtained were less than 10 throughout the tests.

EXAMPLE 5

A padding bath was prepared as follows:

65 parts of methyl hydrogen polysiloxane emulsion was diluted with a small quantity of water and to this was added a solution of 13 parts of sodium bicarbonate and 50 parts of urea in water. 10 parts of "Dispersol" Fast Yellow T300 (Colour Index Disperse Yellow 42) (C.I. 10338) dispersed in 300 parts of water were allowed to stand with occasional stirring before being sieved through a 240 B.S.S. mesh sieve into the padding bath. The bath was made up to 1000 parts with water.

A rainwear fabric comprising 100% polyethylene terephthalate fibers was passed through the padding bath so prepared and squeezed to retain 45% of the bath liquor. The treated fabric was dried at 120° C. and then cured at 200° C. for 90 seconds. Finally the fabric was washed in a 0.2% soap solution at 45° C. and then rinsed with

water until a clear rinse resulted, the fabric then being dried and left for 24 hours before testing.

The resulting dyed fabric was fast to washing and to wet and dry rubbing. It has a spray rating of 100 and proved acceptable when subjected to the Bundesmann tests as referred to in Example 1.

EXAMPLE 6

A padding bath was prepared as follows:

60 parts of methyl hydrogen polysiloxane emulsion was diluted with a small quantity of water and to this was added a solution containing 10 parts of sodium bicarbonate and 50 parts of urea in water. 5 parts of "Dispersol" fast Yellow T300 (Colour Index Disperse Yellow 42) and 2 parts of "Luranol" Blue TR300 (Colour Index Disperse Blue 56) (an anthraquinone dye) dispersed in 300 parts of water were allowed to stand with occasional stirring before being sieved through a B.S.S. 280 mesh sieve into the padding bath. The bath was made up to 1000 parts with water.

A rainwear fabric comprising of 55/45 polyethylene terephthalate cellulose tri-acetate fibers was passed through a padding bath and squeezed to retain 45% of the bath liquor. The treated fabric was dried at 120° C. and aired at 200° C. for 90 seconds. Finally the fabric was washed in a 0.2% soap solution at 45° C. and then rinsed with water until a clear rinse resulted. The fabric was then dried and left for 24 hours before testing.

The resulting dyed fabric was fast to washing and wet and dry rubbing. It had a spray rating of 100 and proved acceptable when subjected to Bundesmann as referred to in Example 1.

What is claimed is:

1. A process for the simultaneous dyeing and water-proofing of fabrics containing mixtures of polyester fibers and cellulose fibers, comprising treating said fabrics in an

aqueous medium comprising at least a methyl hydrogen polysiloxane water-proofing composition, an alkaline catalyst selected from the group consisting of sodium bicarbonate and sodium sesquicarbonate, a dyestuff mixture which dyes each of the fibers in the fabric comprising (1) a disperse dyestuff and (2) a reactive dyestuff in which the reactive component is a cyanuryl chloride, the aqueous medium also containing a cross-linking resin selected from the group consisting of melamine formaldehyde, dimethylolethylene urea, triazone and methylated urea-formaldehyde resins followed by drying the treated fabric and then heat treating the fabric at a temperature of at least 100° C.

2. A process according to claim 1 wherein the heat treatment is carried out at a temperature of 110–220° C. for a time of 15 seconds to 5 minutes.

3. A process according to claim 1 wherein the silicone composition on the finished fabric is from 0.5–5% by weight of the fabric.

4. Fabrics containing polyester fibers and cellulosic fibers treated according to a process claimed in claim 1.

References Cited

FOREIGN PATENTS

242,146 3/1960 Australia.
846,505 Great Britain.

OTHER REFERENCES

Welch: Textile Research J., February 1963, pp. 165–167.

DONALD LEVY, Primary Examiner

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117–139.5; 8–21, 163