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PROCESS FOR DYEING CELLULOSE
TRIACETATE BLENDS

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The present invention relates to the production of reserve, cross dyeing or union colors in fabrics comprising fibers of cellulose esters of low hydroxyl content in blends or combinations with other fibers, particularly cellulose fibers and especially cotton, either by selectively dyeing the cellulose ester fiber of the blend while reserving the other fiber or by dyeing the cellulose ester fiber one shade and the cellulose fiber another shade.

The invention is particularly directed to the continuous treatment of fabrics comprising cellulose esters of low hydroxyl content blended with cotton to provide cross dye effects using continuous treating procedures and equipment available in the cotton dyehouse.

Another object of the invention is the provision of a dyeing process for cellulose esters of low hydroxyl content whereby increased efficiency in dye utilization is achieved and deeper shades obtained.

Other objects of the invention will be apparent from the description and claims which follow. In this description and claims all proportions are by weight unless otherwise indicated.

In accordance with one aspect of the invention a fabric comprising fibers of a cellulose ester of low hydroxyl content such as cellulose triacetate together with another fibrous material such as a cellulose fiber, e.g., cotton, is impregnated with a solution of a cellulose acetate dye dissolved in an aqueous solution of a water-soluble lower alkyl phosphate, e.g., triethyl phosphate. The impregnated fabric is then steamed and thereafter washed to remove excess dye, desirably with hot water and then with soap.

The cellulose esters of low hydroxyl content employed in the process of the invention contain not more than 0.29, preferably zero to 0.12, alcoholic hydroxyl groups per anhydroglucose unit in the cellulose molecules thereof. Best results are obtained by the use of cellulose acetate of very high acetyl value, e.g., an acetyl value of at least 59%, preferably 61 to 62.5%, calculated as combined acetic acid, and hereinafter referred to as triesters. Cellulose triacetate is the preferred cellulose ester. However, other lower aliphatic acid esters of cellulose of low hydroxyl content may be employed. Examples of such esters are cellulose propionate, cellulose butyrate, cellulose acetate-propionate, cellulose acetate-butyrate, cellulose acetate-formate.

Any cellulose fiber may be employed in the fabric blends which are treated in accordance with the invention. While cotton is preferred, other examples of cellulose fibers which may be employed are linen, ramie, sisal, viscose rayon and cuprammonium rayon.

Advantageously, the proportion of cellulose triester in the blend ranges from about 8 to 92% by weight to produce deep shades, although the method of the invention is also operative at higher or lower proportions; preferably the cellulose triester proportion ranges from about 20 to 50% with the balance preferably comprising a major proportion of cellulose fibers.

Animal fibers such as wool and synthetic fibers made, for example, from polyamide, polyester and polyacrylic fibers may also be employed in admixture with cellulose ester fibers and will be reserved to some extent in the treatment. These other fibers are less preferred since, unlike cotton, rayon and other cellulosic fibers, they are

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dyed by the treatment, although less strongly than the cellulose ester fibers.

The concentration of the lower alkyl phosphate may range between about 10 and 30% and preferably from 15 to 25% based on the weight of the dye liquor. The phosphate is of low volatility and the dyebath is non-inflammable.

The temperature of the dyebath is desirably between 175–195° F., but temperatures of 95° F. to boiling may be used. If convenient, the time of immersion of the fabric in the dyebath may be anywhere from instantaneous to one minute. An immersion time from 5 to 15 seconds is preferred. The higher the bath temperature, the shorter is the required immersion time.

The disperse acetate dyes listed in Colour Index, 2nd edition, pages 1659–1742 may be used. These disperse dyes may be used in any concentration up to their maximum solubility in the bath which will vary in individual cases from 2 to 10% by weight. Normally, the disperse dyes are dissolved to provide a dye concentration in the dye liquor of from 0.1–5% by weight. The following disperse acetate dyes, all but the last two of which are of the high temperature slow dyeing type, give particularly good results: Eastone Red N–GLF (CI Disperse Red 35); Eastone Red 2B–GLF; Interchem Brilliant Blue NSP; Interchem Yellow HDLF–40 (PR 625); Lenra Yellow R; Latyl Blue BG; Amacel Yellow CW (CI Disperse Yellow 37); Eastone Yellow 2 RGLF; Amacel Orange BL (CI Disperse Orange 5); Celliton Black BTNA. Other dyes which may be used include Solacet Fast Blue 2 B (CI Acid Blue 14), Solacet Fast Crimson B (CI Acid Red 159), Solacet Fast Green 2G (CI Acid Green 17), Solacet Fast Orange 2 GK (CI Acid Yellow 64) and Solacet Fast Scarlet B (CI Acid Red 53).

The steaming conditions may be anywhere from 5 seconds to 5 minutes at temperature of from about 205° F. to 240° F., but is preferably about 15 to 60 seconds at about 205–225° F.

It is preferred to run the fabric after steaming into boiling water followed by soaping, but these conditions may vary widely.

Dye liquor is picked up during the padding operation on the cellulose fibers as well as the cellulose ester fibers with the dispersed dye being in solution in the dye liquor. During the steaming operation it has been found that the dye picked up by the cellulose fiber is transferred from the cellulose fiber (which is reserved) to the cellulose ester fiber. In this manner, the proportion of dye which is fixed upon the cellulose ester fiber exceeds that proportion which is picked up by the cellulose ester fiber, e.g., by an amount approximately comparable to the additional proportion of dye picked up by the cellulose fiber. In this manner, the dyeing operation of the invention fulfills two valuable functions when applied to fabrics containing both cellulose ester fibers and cellulose fibers. First, the transfer of dye enables deeper shades to be obtained in a single pass. Second, the dye applied to the cellulose fiber is not wasted but is instead utilized in achieving a full depth of shade on the cellulose ester fiber.

The continuous selective dyeing of cellulose ester fibers in fabrics containing these fibers in admixture with other fibers by means of the application of an aqueous solution of dispersed dye followed by steaming is illustrated in the examples which follow:

Example I

Bleached and mercerized 55/45 spun cellulose triacetate/cotton poplin was passed through a dye solution prepared as indicated below:

The solution has a temperature of 205° F.±5° F. The time of immersion of the fabric in the dye solution was 4 seconds, this being achieved by moving the fabric

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at a speed of 60 yards per minute. After impregnation, as aforesaid, the impregnated fabric was steamed at 222° F. for a period of 50 seconds. The steamed fabric was then passed through two boxes of boiling water and then through two additional boxes containing water at 208° F. and containing chip soap and trisodium phosphate. The fabric was then rinsed in hot water and can dried.

The dye solution referred to above was a water solution containing the following components:

- 0.55% Eastman Blue B-GLF
- 0.1% Eastone Red N-GLF
- 0.15% Eastone Yellow 2R-GLF
- 15% triethyl phosphate

The dye solution was produced by dissolving the disperse dye powder in hot (205–210° F.) triethyl phosphate. Water, near boiling temperature, was added to this solution of disperse dye in triethyl phosphate to provide 100 gallons of dye solution containing the proportions set forth above.

As a result of the dyeing procedure described, the cellulose triacetate was dyed a deep brown whereas the cotton remained white.

The cellulose triacetate portion of the fabric described in Example I can be dyed various shades while reserving the cotton or other cellulose fibers contained in admixture with the cellulose triacetate as will be indicated in the examples which follow.

Example II

The process of Example I was repeated employing as the dyebath an aqueous solution containing 1.5% by weight of 2,6-dichloro-4-nitro-4'-(N-methyl-N-hydroxyethyl)-aminoazobenzene and 15% by weight of triethyl phosphate. The cellulose triacetate was dyed a deep reddish orange shade while the cotton remained white.

Example III

The fabric of Example I was dyed using an aqueous solution containing 3% Latyl Brilliant Blue BG and 20% triethyl phosphate, the dye being first dissolved in water and the solution diluted with triethyl phosphate. The fabrics were padded by passing them through the dye solution in a Williams unit maintained at 195° F. The fabric was passed through the Williams unit at 60 yards per minute to provide an immersion time of 12 seconds. The impregnated fabric was steamed at 216° F. for a period of 25 seconds. The dyed fabric was then washed in hot water. The dyed fabric was then rinsed and scoured using soap and trisodium phosphate, the scouring operation being conducted for a period of 30 minutes in a bath maintained at 200° F.

Example IV

The cellulose triacetate component of the fabric of Example I was dyed turquoise using an aqueous solution containing 1% Latyl Brilliant Blue BG, 2% Cellitone Blue-Green BA, 1.2% Interchem Yellow HDLF-40, 1% Conco Inhibitor FR and 20% triethyl phosphate. The conditions of impregnation, steaming and washing were the same as in Example III, the cellulose triacetate component of the fabrics being deeply dyed a turquoise shade while reserving the cellulose fiber contained in the fabric.

Example V

The cellulose triacetate component of the fabric of Example I was dyed tan using an aqueous solution containing 0.37% Eastone Red 2B-GLF, 1.1% Interchem Yellow HDLF-40, 0.12% Lenra Blue RLS and 20% triethyl phosphate. The conditions of impregnation, steaming and washing were the same as in Example III, the cellulose triacetate component of the fabrics being deeply dyed a tan shade while reserving the cellulose fiber contained in the fabric.

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Example VI

The cellulose triacetate component of the fabric of Example I was dyed red using an aqueous solution containing 2% Eastone Red 2B-GLF and 20% triethyl phosphate. The conditions of impregnation, steaming and washing were the same as in Example III, the cellulose triacetate component of the fabric being deeply dyed a red shade while reserving the cellulose fiber contained in the fabric.

Example VII

The cellulose triacetate component of the fabric of Example I was dyed black using an aqueous solution containing 2.14% Interchem Brilliant Blue NSP, 0.5% Eastone Red 2B-GLF, 0.98% Eastone Yellow 2R-GLF, 1% Conco Inhibitor FR and 25% triethyl phosphate. The conditions of impregnation, steaming and washing were the same as in Example III, the cellulose triacetate component of the fabrics being deeply dyed a black shade while reserving the cellulose fiber contained in the fabric.

Further examples illustrating the dyeing procedure of the invention to produce cross dye effects on fabrics composed of cellulose triacetate fibers and cotton fibers are as follows.

Example VIII

The cellulose triacetate component of the fabric of Example I was dyed blue using an aqueous solution containing 78 parts by weight of water, 10 parts of triethyl phosphate, 10 parts of sodium thiocyanate and 2 parts Interchem Brilliant Blue NSP. The fabric was padded from dye liquor (176° F.), after which it was steamed for 30 seconds at 220° F. and then immersed in boiling water for 1 minute, followed by soaping (2 g./l.) for 15 minutes at 185° F., rinsed and dried.

Further formulations for the selective dyeing of cellulose triacetate to produce various shades while reserving the cellulose fiber in admixture therewith are as follows.

Shade—Black:

- 4% Interchem Brilliant Blue NSP
- 1.1% Eastone Red N-GLF
- 1.8% Interchem Yellow HDLF-40
- 20% triethyl phosphate

Shade—Maroon:

- 3% Amacel Rubine IX
- 20% triethyl phosphate

Shade—Brown:

- 1.5% Interchem Brilliant Blue NSP
- .75% Eastone Red 2B-GLF
- 2% Amacel Orange BL
- 20% triethyl phosphate

Shade—Bright Greenish Blue

Shade—Bright Greenish Blue:

- 2% Latyl Brilliant Blue BG
- 20% triethyl phosphate

Shade—Bright Red:

- 1% Eastone Red 2B-GLF
- 0.5% Eastone Red N-GLF
- 20% triethyl phosphate

All of the above formulations are made up to 100 parts with water.

The cellulose fiber component in the blend may be dyed either the same shade as the cellulose triacetate fiber or it may be dyed a contrasting shade. Coloration of the cellulose can be effected either before or after the cellulose triacetate, using known cellulose dyes which are not substantive to cellulose esters, e.g., vat dyes, sulfur dyes, direct dyes, naphthol dyes, reactive dyes, and the like.

The following examples illustrate complete procedures for dyeing both the cotton and cellulose triacetate components of blends:

Example IX

A chambray composed of a wrap of cotton and a filling of cellulose triacetate was padded at the rate of 60 yards

per minute with a 60% by weight pick-up of an aqueous bath maintained at 80° F. and containing per gallon 7 grams of Procion Yellow RS, 0.53 gram Procion Blue 3GS, 3.42 grams Procion Red 2BS, 2 ounces of sodium bicarbonate and 4 ounces of sodium alginate. The fabric was passed through a hot flue at 300° F. with a dwell time of 75 seconds and then entered a second pad maintained at 195° F. The solution in the second pad comprised 15% by weight of triethyl phosphate dissolved in 85% of water containing per gallon 0.92 gram Eastman Blue G-GLF, 0.97 gram of Eastone Red N-GLF and 1.57 grams of Eastone Yellow 2R-GLF. The fabric was then steamed for 1 minute at 220° F., washed for about 2 minutes with boiling water, scoured twice for about 2 minutes each with a boiling solution of soap and trisodium phosphate, rinsed twice with hot water at 160° F., rinsed with water at 120° F. and can dried to produce an apricot-colored union dyeing. The fabric was then heat treated by running at 20 yards per minute over cans maintained in an oven at 435° F. to impart ease of care properties to the cellulose triacetate.

Example X

The product of Example I, after can drying, was padded with 100 gallons of water containing 16 pounds 14 ounces of Indanthrene Brown GAP and maintained at 130° F. The fabric which picked up 60% of its weight of solution was then passed through 100 gallons of water maintained at 110° F. and containing 10 quarts of 50% caustic soda, 10 pounds of sodium hydrosulfite and 10 ounces of Indanthrene Brown GAP double paste. After steaming at 225° F. for 30 seconds, the fabric was passed through two successive Williams units each containing 90 gallons of water at 180° F. to which had been added 8 quarts of 50% caustic soda, 8 pounds of sodium hydrosulfite and 15 ounces of Indanthrene Brown GAP dbl. This cleared the cellulose triacetate. The fabric was then rinsed in cold water and passed through a tank containing 200 gallons of water at 140° F. and having added thereto 2 quarts of 50% hydrogen peroxide and 4 quarts of acetic acid. The fabric was then soaped twice at 200° F., rinsed once in hot water, rinsed in cold water and can dried. The cotton was dyed brown.

The cellulose triacetate-cotton blends can be subjected to varied pre- and post-treatments to which either cotton or triacetate is usually subjected, e.g., mercerization, bleaching heat treatment, and the like. The treatments, of course, should not be so drastic as to damage or materially to alter the character of either of the fiber components. Thus, when mercerizing or vat dyeing to affect the cotton the conditions of time, temperature and pH should be selected so that the cellulose triacetate will not undergo saponification. To this end, a rapid neutralization following the alkaline treatment is desirable. Heat treatment to improve the properties of the cellulose triacetate fibers of the blend can be carried out in conventional manner, e.g., heating in a frame at 390 to 415° F. for about 4 seconds, without injury to the cotton. Such a treatment improves the ability of the cellulose triacetate fibers to resist resin coatings which it may be desired to apply selectively to the cotton; in such event, obviously the heat treatment will precede application of the resin as shown in Example VIII.

The blends comprising mostly cotton will have a cotton hand and can be processed readily on conventional cotton handling equipment. Approximately 50-50 blends, in addition to the color effects noted hereinabove, exhibit many of the ease-of-care properties attributable to cellulose triacetate, such as durable pleating. Blends high in cellulose triacetate have the advantages of rapid drying, etc., exhibited by 100% cellulose triacetate fabrics and are also capable of producing special color effects in accordance with the invention.

It is to be understood that the foregoing detailed description is given merely by way of illustration and that

many variations may be made therein without departing from the spirit of our invention.

Having described our invention what we desire to secure by Letters Patent is:

1. Process for dyeing fabrics constituted by cellulose ester fibers containing at most 0.29 alcoholic hydroxyl groups per anhydroglucose unit in the cellulose molecules thereof in admixture with other fibers selected from the group consisting of cellulose fibers, animal fibers, polyamide fibers, polyester fibers, and polyacrylic fibers, to selectively dye said cellulose ester fibers while reserving said other fibers, comprising impregnating said fabrics with a dye liquor solution of a cellulose acetate dye in water having a lower alkyl phosphate dissolved therein in about 10 to 30% by weight of the solution, steaming said impregnated fabrics, and washing the dyed fabric so produced.
2. Process as recited in claim 1 in which said other fiber is a cellulose fiber.
3. Process as recited in claim 2 in which said cellulose fiber is cotton.
4. Process as recited in claim 1 in which said cellulose ester is a cellulose acetate having an acetyl value of at least about 59% calculated as combined acetic acid.
5. Process as recited in claim 1 in which said lower alkyl phosphate is triethyl phosphate.
6. Process as recited in claim 5 in which said triethyl phosphate is present in said dye liquor in an amount of from 15-25% by weight and said dye liquor contains dissolved therein from 0.1-5% by weight of said dye.
7. Process as recited in claim 1 in which said fabric is impregnated with said dye liquor by immersion of said fabric in said dye liquor for from 5-15 seconds with said dye liquor being maintained at a temperature of from 175-195° F. and is steamed for up to about 60 seconds at a temperature of about 205-240° F.
8. Process for dyeing with a cellulose acetate dye a fabric comprising about 8 to 92% by weight of cellulose ester fibers containing at most 0.29 alcoholic hydroxyl groups per anhydroglucose unit in the cellulose molecules thereof in admixture with other fibers to obtain a deep shade on said cellulose ester fibers in a single pass while consuming substantially all of the dye applied to said fabric without substantially dyeing said other fibers, comprising impregnating said fabric with an aqueous liquor maintained at a temperature of from 95-212° F. and containing said dye in solution together with about 10 to 30% of triethyl phosphate based on the weight of the solution, by immersing said fabric in said liquor for a period up to one minute, steaming said impregnated fabric at a temperature of from 205-240° F. until said dye has been fixed on said cellulose ester fiber and transferred from said other fibers to said cellulose ester fibers, and washing the dyed fabric so produced.
9. Process as recited in claim 8 in which said cellulose ester fiber is cellulose triacetate and said other fiber is a cellulose fiber.
10. Process as recited in claim 9 in which said cellulose triacetate comprises about 20 to 50% by weight of the fiber mixture.
11. Process for dyeing with a cellulose acetate dye a fabric comprising about 8 to 92% by weight of cellulose ester fibers containing at most 0.29 alcoholic hydroxyl groups per anhydroglucose unit in the cellulose molecules thereof in admixture with cotton fibers to obtain a deep shade on said cellulose ester fibers in a single pass while consuming substantially all of the dye applied to said fabric without substantially dyeing said cotton fibers, comprising impregnating said fabric with an aqueous liquor maintained at a temperature of from 95-212° F. and containing said dye in solution together with about 10 to 30% of triethyl phosphate based on the weight of the solution, steaming said impregnated fabric at a temperature of from 205-240° F. until said dye has been fixed on said cellulose ester fiber and transferred from

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said cotton fibers to said cellulose ester fiber, and washing the dyed fabric so produced.

12. Process as recited in claim 10 in which said cotton fibers are dyed prior to impregnation of said cellulose ester fibers.

13. Process as recited in claim 10 in which said cotton fibers are dyed after steaming of said impregnated fabric.

14. Process as recited in claim 10 in which said cellulose ester comprises cellulose triacetate.

15. Process as recited in claim 10, including the further steps of heat treating the fabric and then applying thereto a resin finish for the cotton.

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