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PROCESS FOR DYEING CELLULOSE TRIESTERS WITH DISPERSE ACETATE DYES IN THE PRESENCE OF AN ORGANIC LIQUID

Joseph S. Warner and George C. Ward, Charlotte, N.C., assignors to Celanese Corporation of America, New York, N.Y., a corporation of Delaware

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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 diacetone alcohol, ethoxyethyl acetate, methoxyethyl acetate, butoxyethyl acetate, butoxyethanol, butoxyethoxyethanol, ethyl lactate, glycerol diacetate, glycerol triacetate, acetyl triethyl citrate, ethylene chlorhydrin, butyrolactone or triethanolamine. The wet 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 cellulose fibers, they are dyed by the treatment, although less strongly than the cellulose ester fibers.

Of the enumerated organic liquids in the dyebath, those which are poly-oxygen containing and boil at temperatures in excess of 300° F. are preferred, especially diacetone alcohol employed in a concentration of about 10 to 40% and preferably about 20 to 30%. Ethoxyethyl acetate is preferably employed in a concentration of about 5 to 8%, most preferably about 7%; methoxyethyl acetate desirably about 5 to 25%, preferably about 10 to 20% and most preferably about 15%; butoxyethyl acetate preferably about 5 to 15% and most preferably about 10%; butoxyethanol desirably about 5 to 30%, preferably about 10 to 20% and most preferably about 15%; ethyl lactate desirably about 5 to 30%, preferably about 10 to 20% and most preferably about 15%; butoxyethoxyethanol desirably about 5 to 30%, preferably about 10 to 20% and most preferably about 15%; glycerol diacetate desirably about 5 to 20%, preferably about 6 to 10% and most preferably about 8%; glycerol triacetate preferably about 5 to 8% and most preferably about 7%; acetyl triethyl citrate desirably about 4 to 8%, preferably about 4 to 6% and most preferably about 5%; ethylene chlorhydrin desirably about 5 to 30%, preferably about 10 to 20% and most preferably about 15%; butyrolactone preferably about 10 to 30% and most preferably about 20%; and triethanolamine preferably about 10 to 40% and most preferably about 10%. Where mixtures of these organic liquids are employed, obviously the individual components may be present in less than the indicated preferred amounts while still achieving preferred effects. Where the organic liquid is not soluble in water to the concentration desired, or where an enhanced action is desired, co-solvents such as alcohol, acetic acid, ethylene glycol, the phenol ether of ethylene glycol or glycerine may be employed although at least about 50% and preferably at least about 65% of the dyebath should comprise water. The combined solvent content of course should not be so high as to cause damage to the filamentary material.

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 dye bath 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 sec-

onds to 5 minutes at temperatures 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 weil 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

A poplin fabric comprising a 55/45 intimate blend of cellulose triacetate staple/cotton is padded to a 60% pick-up in a 30% solution of diacetone alcohol in water at 176° F., the solution having dissolved therein 2.86% of Interchemical Brilliant Blue NSP, 0.78% Eastone Red 2BGLF, and 1.3% of Eastman Yellow 2RGLF. Continuously after leaving the pad the wet fabric is immediately steamed at 220° F. for 45 seconds. The thin, non-viscous impregnant is washed out readily by boiling water, the fabric is scoured in soap solution, rinsed in hot water and can dried. The cellulose triacetate fibers are dyed black and the cotton fibers are left practically unstained.

Example II

Little or no staining of the cotton results when the process of Example I is repeated on a twill weave fabric having a warp of 80/20 cotton/cellulose triacetate and a filling of 45/55 cotton/cellulose triacetate. A 2% concentration of Eastman Blue BGLF dyes the cellulose triacetate fibers a medium blue; 2% of Eastone Red NGLF dyes the cellulose triacetate fibers scarlet; 2% of Latyl Brilliant Blue BC dyes the cellulose triacetate fibers turquoise; 1.5% of Eastone Yellow 2RGLF dyes the cellulose triacetate fibers a golden yellow.

Example III

(*a*) The process of Example I is repeated, substituting for the aqueous diacetone alcohol dye solvent a solvent comprising 75% water, 17% diacetone alcohol and 8% of gylcerol triacetate. The cellulose triacetate fibers are dyed black while the cotton is practically unstained.

(*b*) Similar results are achieved using as the dye solvent 70/25/5 water/diacetone alcohol/β-phenoxyethanol.

(*c*) An 8% solution of glycerol triacetate in water containing 0.5% of a non-ionic alkyl aryl polyether alcohol surface active agent, when used as the dye solvent in place of the dyebath of Example I, gives similar results.

(*d*) 80/10/10 water/glycerol triacetate/triethyl phosphate substituted for the solvent in the dye liquor of (*b*) also produces black dyeing of the cellulose triacetate fibers while leaving the cotton practically unstained.

(*e*) An identical process employing as the dye solvent 80/20 water/ethylene chlorhydrin also dyes the cellulose triacetate black while leaving the cotton practically unstained.

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 IV

A chambray composed of a cotton warp and a cellulose triacetate filling, the cotton comprising about 50% of the fabric weight, is 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 R.S., 0.53 grams Procion Blue 3GS, 3.42 grams Procion Red 2BS, 2 ounces of sodium bicarbonate and 4 ounces of sodium alginate. The fabric is passed through a hot flue at 200° F. with a dwell time of 75 seconds and then enters a second pad maintained at 195° F. The solution in the second pad comprises 25% diacetone alcohol, 5% β-phenoxy-ethanol and 70% water containing per gallon 0.92 gram Eastman Blue B–GLF, 0.79 gram of Eastone Red–N–GLF and 1.57 grams of Eastone Yellow 2R–GLF. The fabric is 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 is 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 and finished with melamine-formaldehyde resins for the cotton.

Example V

The product of Example I is padded with 100 gallons of water containing 16 pounds 14 ounces of Indanthrene Brown GAP and maintained at 130° F. The fabric which picks up 60% of its weight of solution is 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 is passed through two successive Williams units each containing 90 gallons of water at 180° F. to which has been added 8 quarts of 50% caustic soda, 8 pounds of sodium hydrosulfite and 15 ounces of Indanthere Brown GAP double paste. This removes the cotton dyes from the cellulose triacetate. The fabric is 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 is then soaped twice at 200° F., rinsed once in hot water, rinsed in cold water and can dried. The cotton is 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.

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., exihibited 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 textile materials of cellulose ester fibers containing at most 0.29 alcoholic hydroxyl groups per anhydroglucose unit in the cellulose molecules thereof, comprising impregnating said textile material with a dye liquor comprising a solution of a cellulose acetate dye in water having about 5 to 8% of ethoxyethyl acetate dissolved therein, steaming the wet impregnated fabric, and washing the dyed fabric so produced.

2. Process for dyeing textile materials of cellulose ester fibers containing at most 0.29 alcoholic hydroxyl groups per anhydroglucose unit in the cellulose molecules thereof, comprising impregnating said textile material with a dye liquor comprising a solution of a cellulose acetate dye in water having about 5 to 25% of methoxyethyl acetate dissolved therein, steaming the wet impregnated fabric, and washing the dyed fabric so produced.

3. Process for dyeing textile materials of cellulose ester fibers containing at most 0.29 alcoholic hydroxyl groups per anhydroglucose unit in the cellulose molecules thereof, comprising impregnating said textile material with a dye liquor comprising a solution of a cellulose acetate dye in water having about 5 to 30% of butoxyethanol dissolved therein, steaming the wet impregnated fabric, and washing the dyed fabric so produced.

4. Process for dyeing textile materials of cellulose ester fibers containing at most 0.29 alcoholic hydroxyl groups per anhydroglucose unit in the cellulose molecules thereof, comprising impregnating said textile material with a dye liquor comprising a solution of a cellulose acetate dye in water having about 5 to 30% of butoxyethoxyethanol dissolved therein, steaming the wet impregnated fabric, and washing the dyed fabric so produced.

5. Process for dyeing textile materials of cellulose ester fibers containing at most 0.29 alcoholic hydroxyl groups per anhydroglucose unit in the cellulose molecules thereof, comprising impregnating said textile material with a dye liquor comprising a solution of a cellulose acetate dye in water having about 5 to 30% of ethyl lacetate dissolved therein, steaming the wet impregnated fabric, and washing the dyed fabric so produced.

6. Process for dyeing textile materials of cellulose ester fibers containing at most 0.29 alcoholic hydroxyl groups per anhydroglucose unit in the cellulose molecules thereof, comprising impregnating said textile material with a dye liquor comprising a solution of a cellulose acetate dye in water having about 5 to 30% of ethylene chlorhydrin dissolved therein, steaming the wet impregnated fabric, and washing the dyed fabric so produced.

7. Process for dyeing textile materials of cellulose ester fibers containing at most 0.29 alcoholic hydroxyl groups per anhydroglucose unit in the cellulose molecules thereof, comprising impregnating said textile material with a dye liquor comprising a solution of a cellulose acetate dye in water having about 10 to 30% of butyrolactone dissolved therein, steaming the wet impregnated fabric, and washing the dyed fabric so produced.

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