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CONTINUOUSLY DYEING AND SAPONIFYING CELLULOSE ACETATES AND BLENDS THEREOF BETWEEN AN IMPERMEABLE BLANKET AND A HEATED ROLLER

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This invention relates to the treatment of textile materials to obtain improved and more efficient methods of dyeing textile materials, and it particularly relates to such processes requiring high temperatures.

Heretofore, the dyeing of fabrics such as cellulose acetate and cellulose triacetate, which require high temperatures in the dyeing process, often involved long and tedious operations resulting in lowered efficiency and increased costs, especially when the fabrics consisted of combinations or blends of different types of fibers.

It is one object of the present invention to overcome the aforesaid disadvantages by providing a process whereby the dyeing of fabrics of the aforesaid type with either developed or dispersed dyes is greatly accelerated by continuous processing of the fabrics during their treatment.

Another object of the present invention is to provide a process of dyeing fabrics comprising cellulose esters, especially cellulose triacetate, whereby dye affinity is greatly improved and streaking and creasing, especially when the fabric is winch dyed, is substantially eliminated.

Another object of the present invention is to provide an effective, continuous process of dyeing cellulose acetate/cotton fabrics with satisfactory union shades utilizing naphthol dyes.

Other objects as well as the various advantages of the present invention will be apparent from the following description and claims wherein all parts are by weight unless otherwise specified.

The processing of the fabrics, in accordance with the present invention, involves the impregnating of the fabric with a treating solution which may contain a swelling agent and/or a solvent and/or a caustic, etc. It may also contain one or more dyestuffs. After being impregnated with the solution, the fabric is passed over a heated drum, generally of stainless steel coated with a non-stick substance such as poly-haloethylene, while enveloping the fabric on the drum with a heated rubber gas- and liquid-tight blanket, whereby a chamber is formed between the drum and blanket for the steam generated by the evaporation of part of the liquid with which the fabric is impregnated. The blanket is separately heated by a separate heated roller which may be referred to as the blanket roller. If the impregnant liquid contains a dyestuff, the fabric is effectively and rapidly dyed during its passage over the drum and under the blanket. If there is no dyestuff in the liquid, the fabric is so affected during the treatment that its dye affinity is increased to permit more effective and rapid dyeing thereafter by standard methods utilizing winches, jigs, etc. Such pretreated fabric is especially resistant to streaking and creasing which otherwise often occurs upon winch dyeing.

The contact time between the fabric and the drum and blanket may be between about 2 seconds and two minutes, depending upon the fabric, type of treatment and speed of the machine. The temperature of the drum and blanket during the period of contact with the fabric may vary between about 140° and 350° F. but, for most purposes, is preferably between about 280° and 320° F.

The process of the invention is advantageously used for developed or dispersed dyeing and may be utilized for

either reserve, union or cross dyeing of fabrics containing combinations or blends of fibers of different types. The dyestuffs used are standard dyestuffs listed in the Colour Index.

In accordance with one aspect of the present invention the fabric containing cellulose triacetate is passed through a bath containing swelling agents and is then heated for short duration at high temperature. The cellulose triacetate so pretreated will dye readily on the winch in even streak-free deep shades, without carrier or accelerant. Aqueous diacetone alcohol preferably of about 15 to 35% concentration is the preferred swelling agent but others, alone or in admixture may also be employed, e.g. up to about 10% of phenyl glycol ether, triethyl phosphate, butyrolactone, lower alcohols such as ethyl alcohol, isopropyl alcohol or butyl alcohol, aromatic alcohols such as benzyl alcohol, lower alkyl phenols such as meta creso, glycol esters and/or ethers such as ethylene glycol monobutyl ether acetate, glycerol esters such as the triacetate, 50% acetic acid, and the like. The subsequent dyeing of the pretreated material can then be effected in known manner even without carriers by jig or winch. Representative dyestuffs suitable for dyeing the cellulose triacetate include: Interchem Brilliant Blue NSP, Interchem Acetate Green Blue ALF–40 (CI, Second Edition Part II, No. 62500).

In accordance with another aspect of the invention the cellulose triacetate is dyed rapidly with developed colors in a continuous pass through the swelling agent bath. The bath also contains a stabilized diazo compound, a coupling component therefor, and an acid liberator which destabilizes the diazo compound when heated so as to permit coupling with the coupling component. The material may thereafter be washed with acid to complete dyeing, followed by rinsing and clearing with a mild bleach such as sodium hypochlorite, if necessary. The process is so effective that it can even be carried out on cellulose triacetate which has previously been heat set.

A preferred dye is that produced from a diazotized amine such as 3-acetamido-4-(di-hydroxyethyl)-amino-4'-amino-azobenzene stabilized with an amine such as diethylamine and ultimately coupled with a naphthol such as β-oxynaphthoic acid. A suitable material sold commercially under the name Eastman Black T contains the stabilized diazotized amine plus the coupling component. This produces excellent black shades which are otherwise difficult to achieve on cellulose triacetate.

The concentration of the stabilized diazo compound plus coupling component in the dyebath will generally range from about 1 to 6%, depending upon the depth of shade desired. The dyebath temperature may vary widely although generally it will be between about 100 and 160° F. Excessively high temperatures will reduce the pH and initiate dyeing in the dyebath rather than on the hot roll, with possible streaking.

The acid liberator is preferably ammonium chloride but other acidic substances such as ammonium sulfate, ammonium acetate, or ethyl tartrate could also be employed. It may be present in about 1 to 5% by weight, larger amounts tending to cause the dyestuff to precipitate from the dyebath. As an acid to complete color development a 4 gram per liter solution of formic acid in water has proven especially suitable although sulfuric, hydrochloric or acetic acids could be used. The concentration may be higher or lower, as required to complete development.

The material treated as described hereinabove may be continuous filaments and/or staple fibers in the form of a yarn, tow, non-woven fabric, or knit or woven fabric. The cellulose triacetate may comprise the treated material in whole or in part, being blended with other

fibrous materials such as cotton, wool, silk, rayon, nylon, polyesters and/or acrylics.

In accordance with a further aspect of the present invention naphthol dyes may be used to obtain very satisfactory union shades on blends of cotton and cellulose acetate, the latter being present either as cellulose triacetate or as secondary cellulose acetate. Heretofore, such dyeing required a long and carefully controlled four-step operation consisting of (1) dyeing the cotton by padding on the naphthol dye, (2) coupling with a fast color salt, (3) loading on a winch and dyeing on a naphthol and a fast color base and (4) diazotizing. In accordance with the present method, there is a simultaneous saponification of the cellulose acetate portion of the fabric and dyeing of the cellulose acetate/cotton blend by adding a higher concentration of sodium hydroxide to the dyebath than is necessary to dissolve the naphthol dyestuff, resulting in a surface saponification of the cellulose acetate, and passing the fabric between a heated drum and blanket, of the type described above, for fixation of the naphthol and saponification of the cellulose acetate. To facilitate the attack of the cellulose acetate a small amount of a swelling agent may be present in the dyebath. The fabric may then be given an optional salt rinse followed by treatment with a diazo component which couples with the naphthol component on the fiber. Representative naphthols include Naphthol ASG (Colour Index Azoic Coupling Component 5—CI, 2nd ed., Part II, No. 37610), Naphthol ASD (Colour Index Azoic Coupling Component 18—CI, 2nd ed., Part II, No. 37520), Naphthol ASSW (Colour Index Azoic Coupling Component 7—CI, 2nd ed., Part II, No. 37565) as well as those on pages 2613 to 2628 of volume II of the Colour Index. Representative diazo components include Fast Scarlet G Salt (Colour Index Azoic Diazo Component 12—CI, 2nd ed., Part II, No. 37105), Fast Blue BB Salt (Colour Index Azoic Diazo Component 20—CI, 2nd ed., Part II, No. 37175), Fast Yellow GC Salt (Colour Index Azoic Diazo Component 44—CI, 2nd ed., Part II, No. 37000) and the fast color salts listed at pages 2571 to 2612 of volume II of the Colour Index.

The invention is described further in the following illustrative examples.

Example I

80 yards of cellulose triacetate taffeta and 80 yards of cellulose triacetate gabardine were padded in an aqueous bath containing 25% diacetone alcohol and 5% phenyl glycol ether at 176° F. The fabrics were passed through the bath and between the drum and blanket of the apparatus described above at a speed to provide a contact or dwell time of 10 seconds while the drum was at a temperature of 280° F. and the blanket roller at 320° F.

The fabrics were then passed through four wash boxes, the first containing water at 203° F., the second containing an aqueous solution of 1 gram per liter of soap at 203° F., the third containing water at 203° F. and the fourth containing water at 149° F.

The gabardine fabric was then dyed on a winch to a charcoal shade along with an untreated piece of the same fabric, using an aqueous dyebath containing by weight 2.9% of a disperse dye composition and, based on the fabric weight, 10% of butyl benzoate.

It was observed that the pretreated gabardine fabric was much bluer and darker in shade than the untreated fabric, the pretreated fabric containing an estimated four times as much dye as the untreated fabric. It was also observed that the pretreated fabric was completely free of winch creases, whereas the untreated fabric had a substantial amount of winch creases. The pretreated taffeta fabric was about twice as heavy in shade as the untreated taffeta and was considerably freer of yarn streaks than the untreated taffeta.

Upon heat setting, the pretreated fabrics changed very little in shade and became only slightly harder in hand. The untreated fabrics, on the other hand, became deeper in shade and quite stiff after heat setting.

The pretreatment produces similar advantages in fabrics dyed on the jig.

Example II

The same process was used as in Example I except that the fabric was a cellulose triacetate/rayon twill. The aqueous pretreating bath contained by weight 25% diacetone alcohol and 5% phenyl glycol ether and was maintained at 176° F.

The fabric was passed through the preheating bath and between the drum and blanket of the apparatus described above at a speed to provide a dwell time of 10 seconds while the temperature of the drum was 280° F. and of the blanket roller was 330° F. The fabric was then washed in boiling water, soaped in an aqueous solution containing 1 gram per liter of soap at 203° F. and then successively rinsed in two separate baths of hot water at 203° F.

After the above pretreatment, the fabric was dyed on a winch to a brown shade using an aqueous dyebath containing by weight 2.31% of a brown disperse dye composition.

The fabric was dyed to the brown shade with approximately half the amount of dyestuff ordinarily required even with the use of an accelerant. It was, moreover, substantially free of winch creases.

Example III

Two pieces of cellulose triacetate sharkskin fabric weighing 37.5 kilograms were run through an aqueous bath containing 25% diacetone alcohol and 5% phenyl glycol ether at 176° F. and then between the drum and blanket of an apparatus such as described above. The temperature of the drum was 285° F. and the blanket roller was 330° F. The dwell time of the fabric between the drum and blanket was 10 seconds. Six gallons of liquor were used indicating a pick-up of 55%.

The fabric was then dyed on a winch to a dull navy shade using an aqueous bath containing by weight 3¾% of a disperse dye composition, the bath being at 185° F.

The resultant fabric had far less creasing than was theretofore possible with winch dyeing of this fabric.

Example IV

Two pieces of the same type cellulose triacetate sharkskin as in Example III were run through an aqueous bath containing 25% diacetone alcohol and 5% phenyl glycol ether at 176° F. and then between a drum and blanket as described above at a speed to give a dwell time of 10 seconds. The temperature of the drum was 280° F. and of the blanket roller 320° F. The fabric was then run through a first wash box containing water at 210.2° F., a second containing a soap solution at 210.2° F., a third containing water at 210.2° F. and a fourth containing water at 140° F. The fabric was then loaded onto a winch and dyed a navy shade using an aqueous dyebath containing by weight 3.5% of a disperse dye composition, 1 gram per liter of an anionic fatty amide sulfate sold under the name Dianol S, 0.75 gram per liter of the neutral sodium salt of a complex condensed aryl organic acid sold under the name Tamol N, and 1.5% of diphenyl ethylenediamine sold under the name Conco Inhibitor FR. The bath was at 185° F.

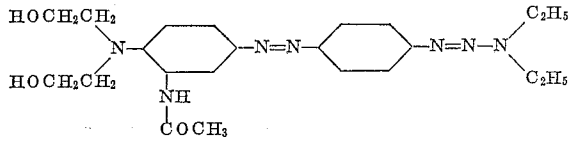
After dyeing, the fabric was dried and heat set at 435° F. and was then finished with an aqueous solution containing 20 grams per liter of dimethylsiloxane plus ortho methyl hydrogen siloxane sold under the name Decetex 104, 5 grams per liter of an emulsion of polyvinyl acetate sold under the name Elvacet, 5 grams per liter of a metal organic salt sold under the name 104 Catalyst and 1 gram per liter of an alkylaryl polyether

alcohol sold under the name Triton X–100. The fabric was then loop cured at 320° F. for 4 minutes and dry filled with an aqueous solution containing 30 grams per liter of a dispersion of colloidal silica sold under the name Siton W–20 and 0.25 gram per liter of dioctyl sodium sulfosuccinate sold under the name Decerosol OT. The fabric was then calendered and decatized.

The finished fabric was entirely free of winch creases and was entirely satisfactory from the standpoint of shaded edges and facing.

Example V

A dyebath was made up which contained 8 gallons comprising 5% ammonium chloride, 25% diacetone alcohol, 5% phenyl glycol ether, 1% benzyl alcohol and 4% of an equimolar mixture of β-oxynaphthoic acid and



the latter being the diethylamine-stabilized product of diazotized 3-acetamido-4-(di-hydroxyethyl) - amino - 4'-amino-azobenzene. The temperature of the bath was maintained at 140° F.

The fabric treated comprised cellulose triacetate/cotton checkster. It was passed through the dyebath and between a heated drum and blanket, of the type described above, at a speed giving the fabric a contact or dwell time of 30 seconds between the drum and blanket. The temperature of the drum was 285° F. and the temperature of the blanket roller was 335° F. After leaving the drum and blanket, the fabric was passed for 1 minute each first through three wash boxes containing 4 grams per liter of formic acid at 140° F. and then through a fourth containing a warm rinse of water at 140° F. The cotton was then cleared by loading the fabric on a jig and washing in a clearing bath containing 2 grams per liter of soap, 0.5 gram per liter of tetrasodium pyrophosphate and 1.5 grams per liter of sodium hydrosulfite at 203° F. for ½ hour.

Example VI

The fabric treated comprised 100% cellulose triacetate and the dyebath comprised 10 gallons of an aqueous solution at 140° F. containing 5% Eastman Black T, 5% ammonium chloride, 25% diacetone alcohol, 5% phenyl glycol ether and 1% benzyl alcohol.

The fabric was passed through the dyebath and between the drum and blanket of the apparatus described above in a manner to provide a contact time of the fabric with the drum and blanket of 30 seconds while the drum was 285° F., after which it was passed through third and fourth wash boxes containing warm water at 140° F.

After the above treatment, the fabric was loaded onto a jig and treated for 20 minutes in an aqueous solution containing 1.5 grams per liter of formic acid. It was then rinsed and thereafter soaped in an aqueous solution containing 1.5 grams per liter of soap flakes and 0.5 gram per liter of sodium hydrosulfite at 194° F.

An excellent black was obtained on the cellulose triacetate, the dyeing having excellent washfastness together with good lightfastness.

Example VII

A black dyeing of the cellulose acetate portion of a cellulose acetate/viscose rayon fabric, with the rayon portion reserved, was obtained as follows:

A 50/50 cellulose acetate/viscose rayon fabric was immersed in an 8-gallon aqueous bath containing 3.5% Eastman Black T, 30% diacetone alcohol and 5% ammonium chloride at a temperature of 149–158° F. The fabric was run through the bath and then it was passed between both the drum and blanket of the apparatus

described above at a speed to give a dwell time of 30 seconds, the temperature of the drum being 240° F. and of the blanket roller 320° F. Then the fabric was washed in aqueous solutions containing 2 grams per liter of formic acid at 194° F. It was then scoured on a winch to remove surface dyestuff and to clear the rayon by using a bath containing 2 grams per liter of soap and 0.25 gram per liter of tetrasodium pyrophosphate at 185° F. for ½ hour and then a bath containing 0.25 gram per liter of tetrasodium pyrophosphate and 0.5 gram per liter of sodium hydrosulfite at 131° F. for 20 minutes. It was then rinsed, extracted and dried.

The acetate fibers were dyed a deep black with the viscose rayon reserved.

Example VIII

A fabric consisting of a warp of filament cellulose acetate and a filling of cotton covered rubber (Lastex) was padded in an aqueous solution containing by weight 3% sodium hydroxide, 3% Naphthol ASD, 2% diacetone alcohol, 0.3% of a cresylic acid mixture sold under the name Mercetex and 0.5% NaCl at a temperature of 180° F. The fabric was then passed between the drum and blanket of the apparatus described above at a speed sufficient to provide a dwell of 15 seconds with the drum being at a temperature of 140° F. and the blanket roller at 240° F. The fabric was then given a five minute rinse in water containing 10 grams per liter of NaCl at 60° F. It was then immediately immersed in a 0.5% solution of Fast Scarlet G Salt at 85° F. for ½ hour, after which it was soaped for 15 minutes at 200° F. in water containing 3 grams per liter of soap and 0.5 gram per liter of soda ash. It was then rinsed and dried.

A bright red shade was obtained on both the cotton and the cellulose acetate fibers. The fabric so dyed had excellent fastness to washing and good fastness to light.

Example IX

Fabric of the same construction as in Example VIII was padded in an aqueous solution containing by weight 3% NaOH, 3% Naphthol AS–G, 2% diacetone alcohol, 0.3% of a cresylic acid mixture sold under the name Mercetex and 0.5% NaCl. The fabric was then passed between the aforesaid drum and blanket to dry in the same manner as in Example VIII, after which it was developed in a 1% solution of Fast Yellow G Salt at 85° F. for ½ hour. It was then soaped for 15 minutes at 200° F. in water containing 3 grams per liter of soap and 0.5 gram per liter of soda ash, after which it was rinsed and dried.

A bright yellow shade was obtained on both the cotton and cellulose acetate fibers with good fastness to washing and light.

Example X

A fabric containing spun cellulose acetate yarn in both warp and filling and a 100% cotton fabric were padded in an aqueous solution containing by weight 3% sodium hydroxide, 3% Naphthol ASSW and 0.5% sulphated ricinoleic acid ester sold under the name Sandozol N at 180° F. and then passed between the drum and blanket described above at a speed to provide a dwell of 15 seconds, the drum being at 280° F. and the blanket at 320° F. The fabric was then rinsed for 5 minutes at 110° F. in an aqueous solution containing 10 grams per liter of NaCl and 0.25 gram per liter of the sodium salt of N-methyl-oleyl taurine sold under the name Igepon T. The dyeing was then completed by treatment for ½ hour at 85° F. in an aqueous solution containing 6% Fast Blue BB Salt.

The cellulose acetate was dyed a deep navy shade with the cotton being somewhat lighter. It was observed that the dyeing had excellent fastness to washing and good fastness to light.

While the invention has been particularly described with reference to dyeing of woven fabric, the filamentary

material may be dyed in the form of knit or non-woven fabrics, tows, yarns, sliver, staple fiber, etc., either alone or in blends with other filamentary materials which will be cross-dyed, union dyed or reserved, depending upon their affinities.

It is to be understood that the foregoing detailed description is merely given 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. A continuous process of dyeing filamentary material composed at least partly of a cellulose ester selected from the group consisting of cellulose acetate and cellulose triacetate, which comprises passing said material through a dye bath containing a dyestuff comprising 1 to 6% by weight of the dye bath of a stabilized diazo compound and a coupling component therefor, said dye bath also contatining 2 to 35% by weight of a swelling agent for at least a portion of said material, 1 to 10% by weight of an acid liberator, and a solubilizer for said dyestuff, subsequently passing said material directly between a heated rotating drum coated with a non-stick substance and a longitudinally moving heated gas- and liquid-tight blanket which is substantially free of any coloring matter when not in contact with said material to provide a contact time between all portions of the material and the drum and blanket of about 5 seconds to 2 minutes, the temperature of the drum and blanket being in the range of about 240 to 335° F., and contacting said material with an acid developer after it leaves the drum and blanket.

2. The process of claim 1 wherein the dyestuff comprises stabilized 3-acetamido-4-(di-hydroxyethyl)-amino-4'-amino-azobenzene and a naphthol coupling component, whereby a deep black shade is obtained.

3. A process of union dyeing a cellulose acetate/cotton blend filamentary material with a naphthol dyestuff which comprises passing said material through a treating bath containing the naphthol dyestuff and a saponifying agent for the cellulose acetate, said saponifying agent being adapted to dissolve said dyestuff in the bath and being present in an amount greater than required to dissolve the dyestuff and sufficient to saponify the surface of the cellulose acetate, subsequently passing the material directly between a heated rotating drum coated with a non-stick substance and a longitudinally moving heated gas- and liquid-tight blanket which is substantially free of any coloring matter when not in contact with said material at a speed to provide a contact time of the material therewith of about 2 seconds to 2 minutes while the drum and blanket are at a temperature of about 140 to 350° F. and then coupling with a fast color salt.

4. The process of claim 3 wherein the bath contains about 3% by weight sodium hydroxide, about 3% by weight of the naphthol dyestuff, about 2% by weight diacetone alcohol and about 0.5% by weight sodium chloride.

5. The process of claim 3 wherein said bath also contains a swelling agent for said cellulose acetate.

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