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DYEING CELLULOSE DERIVATIVES

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This invention relates to the dyeing of organic derivative of cellulose fabrics, and relates more particularly to the dyeing of organic derivative of cellulose fabrics by improved methods comprising mechanical impregnation.

An object of our invention is to increase the speed of dyeing of organic derivative of cellulose fabrics.

Another object of our invention is to obtain more uniform dyeings of organic derivative of cellulose fabrics and to obtain said dyeings more rapidly and economically.

Still another object of our invention is the elimination of overdyed creases, shaded selvedges, endedness, overdyed seam bars and other defects in dyed fabrics.

Other objects of our invention will appear from the following detailed description.

In the coloration of fabrics by means of dye baths it is highly desirable that the depth of shade desired in the fabric be reached in as short an immersion time and with as little handling of the material as possible. In this way, an economical production level may be maintained for the apparatus used in processing the fabrics in the dye plant. When dyeing organic derivative of cellulose fabrics, however, and especially organic derivative of cellulose fabrics of a fairly heavy and/or compact construction, it has been found that these materials are dyed more slowly than other textile materials since they are less absorbent and the dye liquor in the dye bath does not penetrate them as rapidly or as completely as with other textile materials. Many dyestuffs, when applied to organic derivative of cellulose fabrics from aqueous baths, yield dyeings which are far from satisfactory and consequently long periods of immersion and manipulation are usually required in order to dye the fabrics in sufficiently deep, non-fugitive and non-crocking shades.

We have now discovered that organic derivative of cellulose fabrics may be dyed rapidly in deep, level shades if the dyestuffs employed are dissolved in an organic liquid medium comprising a lower aliphatic alcohol and the resulting dye solution applied to the textile material by mechanical impregnation methods followed by a topping operation to desired shade on a winch or jig with an alcohol-free dye bath of suitable composition. This new combination dyeing process is especially applicable to the dyeing of organic derivative of cellulose fabrics of compact construction, such as sharkskins, and fabrics which are closely woven and of fairly heavy structure. By employing this novel process, the fabrics may be dyed to shade in an unusually short period of time. The number of ends the fabric must be given on the winch or jig is reduced considerably and the dyeing operation may be carried out without any danger of cracking the fabric. The resulting dyed fabrics are entirely acceptable commercially and are unusually free of overdyed creases, shaded selvedges, endedness and overdyed seam bars, the difficulties usually arising where the dyeing is carried out entirely on the winch or jig.

Various dyestuffs may be employed. These dyestuffs may be acid dyes which are used for the dyeing of wool and which normally show little or no affinity for organic derivatives of cellulose, or they may be those dyes which are relatively insoluble in water and which are applied to organic derivative of cellulose materials in the form of aqueous dispersions after being specially solubilized. When these dyestuffs are applied to organic derivative of cellulose materials by mechanical impregnation methods in alcoholic solutions and said mechanical impregnation is followed by treatment on a winch or jig in accordance with our process, the resulting dyeings are brilliant and of uniform depth, and level, even, shades may be obtained without difficulty over the entire length of the fabric being processed. The amount of dyestuff dissolved in the alcoholic dye liquor will, of course, vary depending upon the final shade desired in the material being dyed and may be from 0.01% to 6.0% based on the weight of the material. In the dye bath employed for the topping operation, we have found

that only 10% to 15% of this amount of dyestuff need be used to obtain the desired shade.

The lower aliphatic alcohols which are suitable for use in the dye bath may be those alcohols which are solvents for the dyestuffs employed and which exert at least a swelling or softening action on the organic derivatives of cellulose. While we prefer to employ ethyl alcohol in the dye liquor, other alcohols such as methyl alcohol, propyl alcohol, isopropyl alcohol, n-butyl alcohol, secondary butyl alcohol, tertiary butyl alcohol, n-amyl alcohol, secondary amyl alcohol and tertiary amyl alcohol, or mixtures of these alcohols may be used. The amount of the alcohol used in the dye liquor will vary depending on the alcohol or alcohols used, but should comprise at least 70% by weight of the dye bath. Where ethyl alcohol is being used, the dye liquor should preferably contain from about 75% to 85% by weight of the alcohol.

The dye liquor may be applied to the organic derivative of cellulose materials by any convenient method of mechanical impregnation but we preferably employ padding. Thus, a fabric may be passed through a solution of the dyestuff, then between nipping rolls of a padding mangle which act to thoroughly impregnate the fabric with the dye liquor, and, after the fabric is suitably dried as by passing it through a heated chamber, it is then passed to a winch or jig where it is topped to the desired shade with an ancohol-free dye bath of suitable composition. The fabric may be processed through the padding mangle at speeds ranging up to 45 yards per minute and the final shade may be reached by topping with 2 to 12 ends on the winch or jig in from 1 to 6 hours. In the usual dyeing process where fabrics of like construction are dyed directly on a winch or jig without prior padding, shades of like depth may be reached only after processing the fabric for 16 to 40 ends taking from 8 to 20 hours. Even under the latter circumstances a large proportion of the fabrics are commercially unsatisfactory due to various faults, a disadvantage substantially eliminated by our process. It can be seen that not only are the difficulties encountered in prior processes overcome by our novel process, but, in addition, the time necessary to dye a given fabric to shade is shortened considerably. This enables a substantial increase in production to be obtained with only the addition of a padding device to the processing equipment. Not only may production frequently be doubled, but we have even been able to treble the production of certain dyed fabrics on a single winch or jig.

The organic derivative of cellulose comprising the fabrics which may be dyed in accordance with our process are cellulose esters and cellulose ethers. Examples of cellulose esters are cellulose acetate, cellulose propionate, cellulose butyrate and mixed esters, such as cellulose acetate-propionate and cellulose acetate-butyrate, while examples of cellulose ethers are ethyl cellulose and benzylcellulose. Not only may fabrics woven from bright or unpigmented yarns of an organic derivative of cellulose be dyed as described, but unusually desirable results are obtained where fabrics woven of pigment yarns are dyed in accordance with our process. In addition the present process may also be applied to mixed materials containing organic derivatives of cellulose yarns and yarns or fibers of other materials such as cotton, silk, wool or regenerated cellulose. In this way differential dyeings may be obtained where the dyes used have no affinity for the other textile materials present, or union dyeings may be obtained if dyes having an affinity for the cotton, silk, wool or regenerated cellulose are incorporated in the dye liquor used for padding and in the dye bath used in the topping treatment on the winch or jig.

In order further to illustrate our invention, but without being limited thereto, the following examples are given:

Example I

A dye bath is made up with the following composition:

	Parts by weight
Celliton Fast Blue AF conc. (the probable formula of which is 1-methylamino-2-carboxy-amide-4-isopropyl-amino-anthraquinone)	275
Hydroxy-amino-anthraquinone	25
4-halo-2-nitro-4'-alkoxy-diphenyl-amine	35
Ethyl alcohol	75,000
Water	25,000

A cellulose acetate fabric of a heavy, sharkskin weave is scoured on a jig, tenter dried and is then dyed with the dye bath of the above composition. The fabric to be dyed is first passed through a trough containing the dye liquor, maintained at a temperature of 20° to 30° C., at a rate of 20 yards per minute. After moving through the dye bath, the fabric is then passed to the nip of a two roller padding mangle where it is thoroughly impregnated with the liquid. The nip of the rolls of the padding mangle are so adjusted that the fabric retains 65% of its own weight of the dye liquor. From the nip of the padding mangle the fabric is passed to a drying chamber where it is dried by circulating air heated to 95° C. After being dried the fabric is run on a jig through a dilute ammonia solution, rinsed in warm water and is then topped on the jig to the desired shade in 4 hours with the number of ends necessary being about 8. The dye liquor employed for topping is maintained at 80° C. and is of the following composition:

	Parts by weight
Celliton Fast Blue AF conc	27.5
Hydroxy-amino-anthraquinone	2.5
4-halo-2-nitro-4'-alkoxy-diphenyl-amine	3.5
Soda ash	125
Triethanolamine	500
Soap	250
Water	500,000

The resulting fabric is dyed a light blue shade and the fabric is unusually free of faults such as shaded selvedges, endedness, overdyed seam bars and overdyed creases. Without preliminary padding the same shade is reached on the jig alone with a minimum of 16 ends and the entire dyeing operation requires 8 hours.

Example II

A cellulose acetate fabric of sharkskin weave is pad-dyed as described in Example I with a dye bath of the following composition:

	Parts by weight
Celliton Fast Blue AF conc	650
Dinitro-amino-diphenylamine	400
Dihalogenated-4-nitro-benzene-azo-dioxyalkyl-chloraniline	35
Ethyl alcohol	75,000
Water	25,000

After being dried, the fabric is then topped to the desired shade on a jig. The topping dye bath is maintained at 80° C. and is of the following composition:

	Parts by weight
Celliton Fast Blue AF conc	65
Dinitro-amino-diphenylamine	40
Dihalogenated-4-nitro-benzene-azo-dioxyalkyl-chloraniline	3.5
Soda ash	125
Triethanolamine	500
Soap	250
Water	500,000

The resulting fabric is dyed a medium blue shade and is unusually free of dyeing faults. The fabric is topped to shade in 5 hours and the number of ends necessary are 10. Without the preliminary padding the same shade is reached on the jig alone with a minimum of 20 ends and the entire dyeing operation requires 10 hours.

Example III

A cellulose acetate fabric of a twill weave and of the weight usually employed for lining material is pad-dyed as described in Example I with a dye bath of the following composition:

	Parts by weight
Setacyl Direct Discharge Blue G (6-chlor-2,4-dinitro-benzene-azo-2-amino-bis-omega-hydroxyethyl-4-acetylamino-anisole)	3,000
Nitrobenzene-azo-benzene-azo-dioxyalkyl-chloraniline	1,200
Acylamino-benzene-azo-alkyl-hydroxy-benzene	325
Ethyl alcohol	60,000
Water	20,000

After being dried the fabric is then topped to the desired shade on a jig. The topping bath is of the following composition:

	Parts by weight
Setacyl Direct Discharge Blue G (an azo dye)	300
Nitrobenzene-azo-benzene-azo-dioxy-alkyl-chloraniline	120
Acylamino-benzene-azo-alkyl-hydroxy-benzene	32.5
Soda ash	125
Triethanolamine	500
Soap	250
Water	500,000

The fabric is dyed a dark brown shade and the desired shade is reached on the jig in 5 hours with 10 ends being necessary. Without the preliminary padding the entire dyeing operation requires 10 hours with a minimum of 20 ends.

Example IV

A mixed cellulose acetate-wool fabric is pad-dyed as described in Example I with a dye bath of the following composition:

	Parts by weight
Celliton Fast Blue AF	2,200
Dihalogenated-4-nitro-benzene-azo-dioxyalkyl chloraniline	200
Ethyl alcohol	75,000
Water	25,000

After being dried the fabric is topped to shade on a winch with a topping dye bath of the following composition:

	Parts by weight
Celliton Fast Blue AF	330
Dihalogenated-4-nitro-benzene-azo-dioxyalkyl chloraniline	30
Soda ash	750
Soap	1,500
Celascour	9,000
Water	3,000,000

By this treatment only the cellulose acetate in the fabric is dyed a medium blue and the wool is left undyed. The desired shade is obtained by topping on the winch in 4 hours while without a preliminary padding operation the same shade is reached on the winch with a minimum of 12 hours.

Example V

A mixed fabric of cellulose acetate and viscose is pad-dyed as described in Example I with a dye bath of the following composition:

	Parts by weight
Celliton Fast Blue AF	375
Dinitro-amino-diphenylamine	375
Hydroxy-amino-anthraquinone	125
Fastusol Blue LGA (direct cotton dye)	600
Fastusol Yellow LRA (Color Index 349–*a*)	100
Fastusol Scarlet LB (direct cotton dye)	50
Ethyl alcohol	45,000
Water	15,000

After being dried the fabric is topped to shade on a winch with a topping dye bath of the following composition:

	Parts by weight
Celliton Fast Blue AF	37.5
Dinitro-amino-diphenylamine	37.5
Hydroxy-amino-anthraquinone	12.5
Fastusol Blue LGA	60
Fastusol Yellow LRA (Color Index 349–*a*)	10
Fastusol Scarlet LB	5
Soda ash	750
Soap	1,500
Celascour	9,000
Common salt	6,000
Water	3,000,000

The entire fabric is dyed a solid green shade by this treatment. The desired shade is reached on the winch in 5 hours. Without preliminary padding the same shade is reached on the winch alone in a minimum of 12 hours.

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. Method for the coloration of organic derivative of celluose textile materials, which comprises padding such textile material with an aqueous alcoholic solution of a dyestuff, said aqueous alcoholic solution comprising at least 70% by weight of a lower aliphatic alcohol which is a solvent for the dye as the sole organic liquid present, rapidly drying the padded material at an elevated temperature and then topping said textile material to the desired shade by subjecting the material to the action of an aqueous, alcohol-free dye bath of the same dyestuff.

2. Method for the coloration of cellulose acetate textile material, which comprises padding such textile material with an aqueous alcoholic

solution of a dyestuff, said aqueous alcoholic solution comprising at least 70% by weight of a lower aliphatic alcohol which is a solvent for the dye as the sole organic liquid present, rapidly drying the padded material at an elevated temperature and then topping said textile material to the desired shade by subjecting the material to the action of an aqueous, alcohol-free dye bath of the same dyestuff.

3. Method for the coloration of fabrics of comparatively compact construction containing yarns of an organic derivative of cellulose, which comprises padding the fabric with an aqueous alcoholic solution of a dyestuff, said aqueous alcoholic solution comprising at least 70% by weight of a lower aliphatic alcohol which is a solvent for the dye as the sole organic liquid present, rapidly drying the padded fabric at an elevated temperature and then topping said fabric to the desired shade by jig dyeing the fabric with an aqueous, alcohol-free dye bath of the same dyestuff.

4. Method for the coloration of fabrics of comparatively compact construction containing yarns of an organic derivative of cellulose, which comprises padding the fabric with an aqueous alcoholic solution of a dyestuff, said aqueous alcoholic solution comprising at least 70% by weight of a lower aliphatic alcohol which is a solvent for the dye as the sole organic liquid present, rapidly drying the padded fabric at an elevated temperature and then topping said fabric to the desired shade by winch dyeing the fabric with an aqueous alcohol-free dye bath of the same dyestuff.

5. Method for the coloration of fabrics of comparatively compact construction containing yarns of cellulose acetate, which comprises padding the fabric with an aqueous alcoholic solution comprising at least 70% by weight of ethyl alcohol as the sole organic liquid present, rapidly drying the padded fabric at an elevated temperature and then topping said fabric to the desired shade by jig dyeing the fabric with an aqueous alcohol-free dye bath of the same dyestuff.

6. Process for the coloration of organic derivative of cellulose fabrics in medium to deep shades with direct dyeing dyestuffs for organic derivative of cellulose materials, which comprises incorporating the major proportion of the direct dyeing dyestuff by padding the fabric with a solution of the dyestuff in an aqueous liquid medium comprising at least 70% by weight of a lower aliphatic alcohol as the sole organic liquid present in said medium, and then rapidly drying the fabric at an elevated temperature and thereafter incorporating the remainder of the direct dyeing dyestuff by subjecting the fabric to the action of an aqueous, alcohol free dye bath containing the said remainder of the dyestuff.

7. Process for the coloration of cellulose acetate fabrics in medium to deep shades with direct dyeing dyestuffs for cellulose acetate materials, which comprises incorporating the major proportion of the direct dyeing dyestuff by padding the fabric with a solution of the dyestuff in an aqueous liquid medium comprising at least 70% by weight of a lower aliphatic alcohol as the sole organic liquid present in said medium, and then rapidly drying the fabric at an elevated temperature and thereafter incorporating the remainder of the direct dyeing dyestuff by subjecting the fabric to the action of an aqueous, alcohol free dye bath containing the said remainder of the dyestuff.

8. Process for the coloration of cellulose acetate fabrics in medium to deep shades with direct dyeing dyestuffs for cellulose acetate materials, which comprises incorporating the major proportion of the direct dyeing dyestuff by padding the fabric with a solution of the dyestuff in an aqueous liquid medium comprising at least 70% by weight of a lower aliphatic alcohol as the sole organic liquid present in said medium, and then rapidly drying the fabric at an elevated temperature and thereafter incorporating the remainder of the direct dyeing dyestuff by winch-dyeing the fabric with an aqueous, alcohol-free dye bath containing the said remainder of the dyestuff.

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