

UNITED STATES PATENT OFFICE

2,380,503

DYEING

Cyril M. Croft and Walter H. Hindle, Cumberland, Md., assignors to Celanese Corporation of America, a corporation of Delaware

No Drawing. Application November 15, 1941,
Serial No. 419,290

9 Claims. (Cl. 8—59)

This invention relates to the dyeing of organic derivative of cellulose textile materials and relates more particularly to the dyeing of organic derivative of cellulose textile materials by mechanical impregnation methods.

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

Another object of our invention is to obtain more uniform dyeings of organic derivative of cellulose textile materials.

A further object of our invention is to obtain said dyeings more economically with increased color yield from the dye bath.

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

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

In the coloration of textile materials by means of dye baths it is an important desideratum that the depth of shade desired in the textile material be reached with as short an immersion time and as little handling of the material as possible. When dyeing organic derivative of cellulose textile materials, however, 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 other textile material. Many dyestuffs when applied to organic derivative of cellulose materials from aqueous baths yield dyeings which are far from satisfactory from the standpoint of depth of coloration and consequently long periods of immersion and manipulation are usually required in order to dye the textile materials in sufficiently deep, non-fugitive shades.

We have now discovered that organic derivative of cellulose textile materials may be dyed rapidly in deep, uniform, level shades by dissolving the dyestuffs in an organic liquid medium comprising an aqueous solution of a lower aliphatic ketone, such as acetone or methyl ethyl ketone, but preferably acetone, and the resulting dye solution applied to the textile material by mechanical impregnation methods followed, when dyeing fabrics, by a topping operation to desired shade on a winch or jig with a second dye bath of suitable composition and free of any ketone. When coloring staple fiber, the topping step may be dispensed with. By employing our novel process, substantial increases in the rate of production are made possible and satisfactory dyeings may be obtained particularly in the case of fabrics of comparatively compact construction, such as

sharkskins, and fabrics which are closely woven and of fairly heavy structure, with a minimum manipulation of the materials and a low concentration of the dyestuffs in the dye bath.

5 In carrying out our invention various dyestuffs may be employed. These dyestuffs may be acid dyes which are used for the dyeing of wool and 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 normally applied to organic derivative of cellulose materials in the form of aqueous dispersions after being suitably solubilized. When these dyestuffs are applied to organic derivative of cellulose textile materials by mechanical impregnation methods in aqueous lower aliphatic ketone solutions and said mechanical impregnation is followed by treatment on a winch or jig in accordance with our process, the resulting dyeings are of uniform depth and brilliance and may be obtained with relatively short immersion times. The amount of the dyestuff dissolved in the dye liquor will vary depending upon the final shade desired in the material which is being dyed and may be from as low as 0.01% up to 4.0%, based on the weight of the material to be dyed, but at every concentration, a very high color yield is obtained.

The organic liquid medium employed comprises, as stated, an aqueous solution of a lower aliphatic ketone, such as acetone or methyl ethyl ketone and the amount of ketone present in the solution employed should preferably be at least 20% by weight, but in the case of acetone, the solution may contain up to 50% by weight. We have found that the desired results may be obtained when acetone is present in solution in amounts of from 35 to 50%, optimum results being obtained when the solution contains about 40%. However, in the case of methyl ethyl ketone, the solution may contain from 18 to 23% by weight.

The dry liquor may be applied to the organic derivative of cellulose textile materials by any convenient method of mechanical impregnation, padding, however, being preferable. Thus, as a continuous process, a fabric may be unwound from a roll, passed through a solution of the dyestuff, then led between nipping rolls of a padding mangle which are adjusted to permit the desired amount of dye liquor to be retained in the fabric, and, after the fabric is suitably dried as by passing it through a heated chamber, it may then be passed to a winch or jig where it is topped to the desired shade with a second dye bath. The second dye bath is one which is free of ketone and of the usual composition, that is, an aqueous

bath containing the desired dyes and dispersing agents. The period of immersion of the fabric in the dye liquor is relatively short and may be from 2 to 10 seconds. If desired, the fabric may be processed through the padding mangle at speeds ranging up to 45 yards per minute. The final shade may be reached by topping with 2 to 12 ends on the jig or winch in from 2 to 6 hours. In the usual dyeing process where fabrics of like construction are dyed directly with a dye bath of the usual composition 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.

Not only is our process applicable to the dyeing of fabrics, but especially desirable results are obtained when organic derivative of cellulose staple fiber in the form of a combed top or a carded or drawn sliver is dyed in this way. Not only may more uniform dyeings be obtained but increased production may be achieved along with a reduction in the amount of dyestuff necessary in order to obtain a desired shade. By carrying out the dyeing operation in accordance with our invention, the required shade for the material may be obtained by test samples prior to running the material through the dye bath, thereby eliminating the necessity for adding additional color while the fabric or staple fiber is dyeing. The level of liquid in the dye bath is maintained by the addition thereto from time to time of dye liquor of the same composition as that initially employed to replace that portion of the dye liquor carried out by the fabric. Consequently, there is no need to drop incompletely exhausted dye baths when the final shade is obtained as in conventional methods of dyeing, this source of loss or waste of color being eliminated. When dyeing staple fibers in the form of a combed top or carded or drawn sliver, a large number of ends may be run through the dye bath at the same time and padded by a single pad, thus greatly increasing the rate of production of dyed staple without additional equipment. The time of immersion of the top in the dye liquor prior to padding may be from 2 seconds to 2 minutes. After padding, the dyed top or sliver is subjected to rapid drying. As stated above, when dyeing staple fibers, the desired shade may be reached in the ketone-containing dye bath without resorting to any topping operation in a second dye bath.

The dyed textile materials in the form of fabrics or tops may be dried by passing said materials through a chamber through which there is circulated air heated to temperatures up to about 95° C. for quite rapid drying and heated to say 80° C. to 90° C. for less rapid drying. Where the particular concentration of acetone used exerts a marked swelling action on the organic derivatives of cellulose it is advantageous that the materials be dried rapidly.

The organic derivative of cellulose fabrics, tops, slivers, yarns or other textile materials which may be dyed in accordance with our process may be cellulose esters or 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 benzyl cellulose. The materials may be mixed materials containing

organic derivatives of cellulose and yarns or fibers of other materials such as cotton, silk, wool or regenerated cellulose and in this way differential dyeings may be obtained where the dyes used have no affinity for the other textile materials present. Especially valuable differential dyeings or cross-dyed effects may be obtained on fabrics containing mixed cellulose acetate and wool yarns when suitably solubilized, dispersed dyes of the "SRA" type are used. The cellulose acetate fibers may be dyed fairly heavy shades while the wool fibers remain uncolored and unstained. If desired, the wool fibers may later be dyed with dyestuffs which have affinity for the wool or the wool may be left uncolored.

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

Example I

A dye bath having the following composition is made up:

	Parts by weight
Acetone	30,000
Water	42,500
1-methylamino-4-isopropylamino-2-carboxyamide-anthraquinone	1,750
Di-halogenated-4-nitro-benzene-azo-di-oxyalkyl-chloraniline	600

A cellulose acetate fabric of a sharkskin weave is passed into a dye bath or trough containing the above dye liquor maintained at a suitable level and beneath a roller submerged therein. The fabric is passed continuously through the dye bath which is maintained at 25° 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 liquor. The nip of the rolls of the padding mangle are so adjusted that the fabric retains 60% of its own weight of the dye liquor. From the nip of the padding mangle the fabric is passed into a drying chamber in which air heated to 92° C. is circulated. After being dried, the fabric is rinsed in warm water and then topped to the desired shade on a jig in 4 hours with an acetone-free dye bath, the number of ends necessary to reach the shade on the jig being about 6. The fabric is dyed a medium blue shade. The dye bath employed for topping the fabric is maintained at 80° C. and is of the following composition:

	Parts by weight
1-methylamino-4-isopropylamino-2-carboxyamide-anthraquinone	250
Di-halogenated-4-nitro-benzene-azo-di-oxyalkyl-chloraniline	400
Soda ash	125
Triethanolamine	125
Soap	250
Water	500,000

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
Acetone	30,000
Water	42,500
1-methylamino-4-isopropylamino-2-carboxyamide-anthraquinone	375
Di-halogenated-4-nitro-benzene-azo-di-oxyalkyl-chloraniline	400
Dinitro-amino-diphenylamine	400

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
1-methylamino-4-isopropylamino-2-carboxyamide-anthraquinone-----	35
Di-halogenated-4-nitro-benzene-azo-di-oxyalkyl-chloraniline-----	45
Dinitro-amino-diphenylamine-----	45
Soda ash-----	125
Triethanolamine-----	125
Soap-----	250
Water-----	500,000

The resulting fabric is dyed a dark tan shade and is unusually free of dyeing faults. The fabric is topped to shade in four hours and the number of ends necessary are 8.

Example III

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

	Parts by weight
Acetone-----	30,000
Water-----	42,500
1-methylamino-4-isopropylamino-2-carboxyamide-anthraquinone-----	100
Hydroxy-amino-anthraquinone-----	100
Dinitro-amino-diphenylamine-----	600

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

	Parts by weight
1-methylamino-4-isopropylamino-2-carboxyamide-anthraquinone-----	10
Hydroxy-amino-anthraquinone-----	20
Dinitro-amino-diphenylamine-----	150
Celascour-----	25,000
Water-----	5,000,000

The dye bath is maintained at 65° C. By this treatment only the cellulose acetate in the fabric is dyed a light tan shade and the wool is left undyed. The desired shade is obtained by topping on a winch for four hours.

Example IV

Cellulose acetate staple fiber, in the form of a top or sliver, is padded with a dye bath of the following composition:

	Parts by weight
Acetone-----	30,000
Water-----	42,500
Wool Blue GL (Color Index No. 833)-----	500

This treatment gives a medium shade of blue on the staple fiber and there is no need for topping the sliver in a second dye bath to reach the desired shade.

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. Method for the coloration of textile materials, which comprises mechanically impregnating a textile material made of or containing an organic derivative of cellulose with a solution of an organic derivative of cellulose dyestuff in an organic liquid medium comprising an aqueous solution of a lower aliphatic ketone as the sole organic liquid present and in a concentration sufficient to have a swelling effect on

said textile material, rapidly drying the impregnated 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 dyebath of the same dyestuff and having no swelling action on the said material.

2. Method for the coloration of textile materials, which comprises mechanically impregnating a textile material made of or containing an organic derivative of cellulose with a solution of an organic derivative of cellulose dyestuff in an organic liquid medium comprising an aqueous solution of acetone as the sole organic liquid present and in a concentration sufficient to have a swelling effect on said textile material, rapidly drying the impregnated 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 acetone-free dyebath of the same dyestuff and having no swelling action on the material.

3. Method for the coloration of textile materials, which comprises mechanically impregnating a textile material made of or containing an organic derivative of cellulose with a solution of an organic derivative of cellulose dyestuff in an organic liquid medium comprising an aqueous solution of methyl ethyl ketone as the sole organic liquid present and in a concentration sufficient to have a swelling effect on said textile material, rapidly drying the impregnated 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, ketone-free dyebath of the same dyestuff and having no swelling action on the material.

4. Method for the coloration of textile materials, which comprises mechanically impregnating a textile material made of or containing cellulose acetate with a solution of a cellulose acetate dyestuff in an organic liquid medium comprising an aqueous solution containing from 35 to 50% by weight of acetone as the sole organic liquid present, rapidly drying the impregnated 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, acetone-free dyebath of the same dyestuff and having no swelling action on the material.

5. Method for the coloration of fabrics, which comprises padding a fabric of comparatively compact construction made of or containing yarns of an organic derivative of cellulose with a solution of an organic derivative of cellulose dyestuff in an organic liquid medium comprising an aqueous solution containing from 35 to 50% by weight of acetone 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 subjecting the material to the action of an aqueous, acetone-free dyebath of the same dyestuff and having no swelling action on the material.

6. Method for the coloration of fabrics, which comprises padding a fabric of comparatively compact construction made of or containing yarns of cellulose acetate with a solution of a cellulose acetate dyestuff, in an organic liquid medium comprising an aqueous solution containing from 35 to 50% by weight of acetone 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 a second aqueous dyebath free of acetone and having no swelling action on the said material.

7. Method for the coloration of fabrics, which comprises padding a fabric of comparatively compact construction made of or containing yarns of cellulose acetate with a solution of a cellulose acetate dyestuff, in an organic liquid medium comprising an aqueous solution containing from 35 to 50% by weight of acetone 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 a second aqueous dyebath free of acetone and having no swelling action on the said material.

8. Method for the coloration of fabrics, which comprises padding a fabric of comparatively compact construction made of or containing yarns of cellulose acetate with a solution of a cellulose acetate dyestuff, in an organic liquid medium comprising an aqueous solution containing

about 40% by weight of acetone 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 a second aqueous dyebath free of acetone and having no swelling action on the said material.

9. Method for the coloration of fabrics, which comprises padding a fabric of comparatively compact construction made of or containing yarns of cellulose acetate with a solution of a cellulose acetate dyestuff, in an organic liquid medium comprising an aqueous solution containing about 40% by weight of acetone 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 a second dyebath free of acetone and having no swelling action on the said material.

CYRIL M. CROFT.
WALTER H. HINDLE.