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METHOD OF MANUFACTURING MULTICOLORED CELLULOSE FABRICS WITH INDOCARBON BLACK SULFUR DYE

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This invention relates to methods of dyeing textiles, and, more particularly, to methods of sequentially dyeing textiles, otherwise known as overdyeing. The invention is also directed to dyed textiles obtained by the novel dyeing methods described herein.

Heretofore known dyed textiles are prone to loss of color or shade, for example, by discharging or bleeding, when in contact with such reducing and dissolving chemicals as are employed during subsequent overdyeing or topping operations with sulfur dyestuffs or vat dyestuffs. These common characteristics or pre-dyed textiles have severely limited their use in instances where they are constructed, as by interweaving, knitting, or interspinning, with dyeable textiles and then subjected to dyeing conditions employed in dyeing with fast colors, e.g., vat or sulfur dyes, which require reducing and/or dissolving chemicals for their application, in order to color the dyeable textiles with a different color and thus provide multicolored textiles.

In making multicolored textiles, by the overdyeing process, dyed yarns, for example, are interwoven with yarns which are prepared for dyeing to form a fabric comprising dyed yarns and dyeable yarns. The fabric is then overdyeed in order to color the dyeable yarns. When employing yarns or fibers dyed with sulfur dyes in the customary manner and subsequently dyeing the interwoven dyeable yarns with a fast color, e.g., vat or sulfur dyes, which require reducing and dissolving agents, the pre-dyed yarns usually discharge and/or bleed severely onto the dyeable yarn during the subsequent dyeing operation, thus imparting an undesirable color thereto and usually an unsightly appearance to the fabric. The pre-dyed yarns also usually lose color during the subsequent dyeing operation, thus spoiling the appearance of the overdyeed fabric.

It is, therefore, an object of this invention to provide dyeing methods wherein a textile pre-dyed with a sulfur dyestuff is subjected to reducing mediums under such conditions as are employed in dyeing with vat or sulfur dyestuffs without causing objectionable bleeding or alteration of the original colors of the pre-dyed textile.

Another object is to provide multicolored textiles and methods for making them employing sulfur dyestuffs in the pre-dyeing operation and vat dyestuffs and/or sulfur dyestuffs in the subsequent or overdyeing operation.

Another object is to provide multicolored textiles and methods for making them from sulfur dye-impregnated textiles and other dyeable textiles wherein objectionable discharging and/or bleeding of the sulfur dye of the already dye-impregnated textiles onto the dyeable textiles is avoided during overdyeing with fast colors which require reducing and/or dissolving chemicals for their application.

Another object is to provide multicolored fabrics and methods of making them wherein a fabric constructed of sulfur color-predyed yarns and other, dyeable yarns is subjected to dyeing with vat or sulfur dyestuffs without causing objectionable alteration or bleeding of the original colors of the pre-dyed yarns.

Another object is to provide patterned or multicolored fabrics having sharp color delineations by dyeing in the piece.

Other objects and advantages of the invention are more readily apparent from the following description.

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This invention in its broad aspects involves the sulfur color-dyeing or vat dyeing of a dyeable textile in combination with a sulfur color-dyed textile, the sulfur color of which has been pre-treated in at least an intermediate leuco form thereof with a diazotized amine or a corresponding color salt, herein collectively referred to as diazo compounds. In more detail, an embodiment of my method involves the steps of treating sulfur color-dyed cellulosic yarns, wherein the sulfur color as applied to the yarns is in at least an intermediate leuco form, with a diazo compound; weaving said treated, sulfur color-dyed yarns with undyed yarns to form a fabric; and then dyeing the undyed yarns with a sulfur dye or a vat dye in the usual manner to form a multicolored fabric.

The sulfur color-dyed textile to be employed in the overdyeing operation is obtained in any suitable manner, and is conveniently obtained by conventional methods of sulfur dyeing, provided that the sulfur color as applied to the textile is treated in at least an intermediate leuco form with the diazo compound. The phrase "at least an intermediate leuco form" includes the substantially full leuco form or an intermediate but still reactive stage, between the substantially full leuco and the substantially fully developed state.

In general, the treatment with a diazo compound is carried out in basically the same manner as described in United States Patent Numbers 1,879,727; 1,874,099 and 1,875,446. Sulfur color-dyed textiles wherein the leuco sulfur color as applied to the textile has been oxidized to an intermediate but still reactive stage, methods of preparing such dyed textiles and treatments thereof with diazo compounds are described in my copending applications Serial Numbers 828,689 and 828,693, filed concurrently herewith, the latter now being abandoned. Improved methods of treating sulfur dyes as applied to textiles while in the substantially full leuco or an intermediate leuco stage with diazo compounds are described in my copending application, Serial Number 828,690, filed concurrently herewith. The treated sulfur color-dyed textiles can also be obtained by oxidizing the substantially full leuco form to an intermediate stage, treating with the diazo compound, re-reducing in a suitable manner, e.g., by any convenient means employing sodium hydrosulfite or sodium hydrosulfite in combination with sodium bicarbonate, and treating again with the diazo compound. This sequence of steps can be repeated, if desired, until the treated sulfur color, as applied to the textile, is almost entirely insoluble in reducing and dissolving agents generally used in sulfur and vat dyeing.

The textile to be overdyeed is constructed from treated, sulfur color-dyed textiles and other dyeable textiles to be dyed a different color during subsequent overdyeing, in any suitable manner, e.g., by weaving, knitting, spinning or the like.

The overdyeing with sulfur colors or vat colors is carried out by any suitable procedure as by conventional dyeing procedures employing sodium sulfide, sodium hydrosulfite or formaldehyde sulfoxylates, e.g., zinc or sodium formaldehyde sulfoxylates, as reducing agents in the presence of the necessary dissolving chemicals. Such dissolving chemicals can be alkaline as in the case of caustic soda, sodium carbonate or the like. The overdyeing step can be conducted by applying reduced and dissolved dyestuffs, or dye pigments can be first applied and then treated with the necessary reducing agents and dissolving chemicals in any suitable manner in order to affix the dyestuff.

The following example is presented to illustrate a procedure for obtaining treated sulfur color-dyed yarns, preliminary to interweaving with undyed yarns to form a fabric for subsequent overdyeing.

Example 1

A 525 pound load of 30/1 cotton yarn was wetted-out in the usual manner in a production yarn dyeing machine having a liquor ratio of 10 to 1. An aqueous solution of sodium sulfide flakes (3% based on the weight of yarn) and 2% of a sequestering agent, e.g., Perma Kleer, were added to the machine and circulated through the yarn for 10 minutes at 180° F. The dye, 35% SoDyeCo Fast Liquid Black G (New Color Index No. Sulfur Black 11), was then added to the machine in two portions on the "outside-in" ten minutes apart. The dyebath was circulated alternately for 7 minutes "outside-in" and for 3 minutes "inside-out," thus providing a 10-minute cycle. The dyebath was circulated in this manner for 20 minutes at 180° F., after which time 30 g./l. common salt were slowly added on the "outside-in" portion of the cycle. The dyebath was circulated in this manner for an additional 20 minutes at 180° F., after which time it was drained and a cold running water wash was applied with water entering through the expansion tank, passing through the yarn, and then passing out through the drain. The cold water running wash was continued for 40 minutes.

An aqueous solution containing 1% sodium chlorite based on the weight of yarn was circulated in the above-described cycles for 30 minutes at 90° F. Thereafter, the solution was drained and the yarn was washed with running water for 20 minutes.

A solution of diazotized 2,5-dichloroaniline (Fast Scarlet GG Base) prepared from 2.5% 2,5-dichloroaniline in the manner described below was added from the "inside-out" and circulated for 20 minutes in the above-described cycles. The solution then was drained and the yarn was washed for 20 minutes with a running wash. A deep black shade was obtained.

The diazotized 2,5-dichloroaniline was prepared by first diluting 1.25% Triton X-100 with water and adding 12.5% of 32% aqueous hydrochloric acid and ice (the total amount of ice and water initially added to dilute the Triton X-100 being about 70%). Next, 5% of a 50% Scarlet GG Base solution was added while stirring and 1.5% sodium nitrite was immediately and quickly added with stirring. Occasional stirring was continued for about 15 minutes at 40° F. Prior to use, 0.9% sulfamic acid and an anti-foam agent were added to the diazotized amine.

After treatment with the diazotized base and the running water wash, the yarn was soaped in the usual manner with 13% low titer soap (2.6% soap solids), 2.6% soda ash, and 0.3% Igepon T-33 (sodium oleyl taurine) at 190° to 195° F. for 20 minutes. The yarn was then finally rinsed and 2% of a softener was applied. The yarn thus dyed and treated had a deep black shade.

A load of 40/1 cotton yarn was dyed and treated in the same manner as described above.

The 40/1 black dyed yarns were slashed with a starch sizing material in the usual manner to facilitate weaving. Employing the 40/1 black dyed and treated cotton yarn and 40/1 white yarn as warp and the 30/1 black dyed and treated cotton yarn and 30/1 white cotton yarn as filling, a black and white checkered fabric having 1/4" squares was woven. A chambray was woven employing all white cotton yarns as filling and all black yarns dyed and treated as described above as warp. Another patterned fabric containing alternate ends of black and white was woven employing all white cotton yarns as filling and white cotton yarns alternating with black yarns dyed and treated as described above as warp. The fabrics were then singed, desized, hydrogen peroxide bleached, mercerized and scoured, thus preparing them for dyeing.

The following examples are presented to illustrate the over dyeing procedure.

Example 2

Pieces of the black and white checkered fabric, the

chambray, and the other patterned fabric were dyed with vat dyes, as listed below, in the following manner:

The piece of woven fabric was padded at 160° F. with a padding dispersion containing 25 g./l. of a vat dye and 5 g./l. of a dispersing agent, Nekal NF (alkylarylsulfonate in an organic solvent). Having picked up 70% of its weight of the padding dispersion, the fabric was dried in an oven and then padded once through a reducing formulation containing 40 g./l. caustic soda, 40 g./l. sodium hydrosulfite and 50 cc./l. of the above-described padding dispersion. The fabric was then immediately run into a steamer unit maintained at 212° F. where it remained for 30 seconds. Then the dyed fabric was cold-rinsed, oxidized with 1% hydrogen peroxide in aqueous solution for approximately five minutes at 150° F. and soaped for 3 minutes at 195° F. with a soap solution containing 5 g./l. soap and 1 g./l. Igepon T-33, thus simulating a continuous pad-steam dyeing operation.

The white yarns in the patterned fabrics were colored in accordance with the color of the particular vat dye employed and the black, sulfur color-dyed yarns in the patterned fabrics remained black, presenting a sharp color contrast. The black dyed yarns of all the fabrics remained black and did not bleed noticeably into the adjoining colored areas. The fabrics thus dyed can be finished in any manner desired.

Vat dyes (the Part 1, New Color Index No. being given in parentheses) employed in the above-described examples were Cibacron Blue GF Double Paste (Vat Blue 6, C.I. 1113), Ponsol Jade Green Double Paste (Vat Green 1, C.I. 1101), Indanthrene Red FBB Double Paste (Vat Red 10, Pr 296), Veranthrene Brilliant Orange GR Powder (Vat Orange 7), Ponsol Yellow 5GLL Paste (Vat Yellow 22), and a mixture of 6 g./l. of Ponsol Yellow 5GLL Paste, and 1.3 g./l. Ponsol Golden Orange 3G Paste (Vat Orange 15, Pr 290) totaling 7.3 g./l. of vat dye instead of the 25 g./l. specified above.

Example 3

For demonstrating the adaptability of the over dyeing method employing the customary "pad jig" procedure, a 13 gram piece made up of a swatch of the black and white checkered fabric, a swatch of the black and white chambray fabric and a swatch of the black and white patterned fabric having alternating ends of black and white, was impregnated by passing it through a padding dispersion of 25 g./l. Ponsol Jade Green Double Paste and 5 g./l. Nekal NF and squeezed to provide a pick-up of 70% of its own weight. Then the fabric was loosely rolled while still wet and immersed into 200 cc. of a solution containing 20 g./l. solid caustic soda, 20 g./l. sodium hydrosulfite and 50 cc./l. of the above-described padding dispersion. The piece was moved in the solution for 30 minutes at 160° F. The piece was then removed, rinsed with cold water, oxidized with hydrogen peroxide and soaped for 10 minutes at 195° F. in a solution of 5 g./l. soap and 1 g./l. Igepon T-33.

The white yarns of the swatches thus dyed were colored a heavy jade green and the black, sulfur-dyed yarns of the swatches thus dyed remained black, presenting a sharp color contrast. The black dyed yarns of the swatches remained black and did not bleed noticeably into the adjacent green colored areas. The fabrics thus dyed can be finished in whatever manner desired.

Cotton yarns dyed with such other sulfur colors as Sulofgene Brilliant Green J, SoDyeSul Liquid Green NJ, and Sulfogene Brown CL4R, then treated with a diazotized base, disposed adjacent to white fibers and subjected to a reducing medium obtained with sodium hydrosulfite by a procedure similar to that hereinbefore described provided textiles wherein no objectionable bleeding of the sulfur color of the dyed yarns or objectionable loss of color of the sulfur color-dyed and treated yarns adjacent thereto had occurred. Also, cotton yarns dyed with sulfur colors from the above-listed sulfur col-

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ors and treated with such other diazotized amines as diazotized o-chloroaniline (Fast Yellow GC Base), diazotized m-chloroaniline (Fast Orange GC Base) and Fast Scarlet Salt GGN (color salt corresponding to diazotized Fast Scarlet GG Base) when disposed adjacent to white fibers and subjected to a reducing medium obtained with sodium hydrosulfite in a manner similar to that described above, provided textiles wherein there is no objectionable loss of original shade of the sulfur color-dyed and treated yarns and no objectionable bleeding of the sulfur color of the dyed yarns.

The leuco sulfur dye as applied to the yarns when treated directly with the diazo compound, i.e., without treatment with sodium chlorite or other oxidizing agent prior to treatment with the diazo compound, when constructed with dyeable yarns, also provide textiles wherein no objectionable loss of color of the dyed and treated yarns and no objectionable bleeding of the sulfur color of the dyed yarns, occurs during exposure to reducing medium.

Overdyeing can be conducted in the manner described in Examples 2 and 3 employing, however, a sulfur dye, such as, for example, SoDyeSul Liquid Yellow ECF (New Color Index designation, Sul Yellow 4; Color Index No. 955) or SoDyeSul Liquid Blue 3GCF (New Color Index designation, Sul Blue 9; Color Index No. 957) in place of the vat dyes specified.

What is claimed is:

1. An improved method of manufacturing multicolor dyed cellulose fabric, said fabric being particularly char-

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acterized in that the colors do not noticeably bleed into each other, comprising the steps of immersing a first cellulosic yarn into a dyebath containing the fully reduced form of Indocarbon Black sulfur dye, oxidizing said dyestuff to the intermediate stage of oxidation, treating said yarn with a diazotized amine, rinsing the yarn, soaping the yarn under alkaline conditions and again rinsing the yarn, forming said first yarn and a second undyed cellulosic yarn into a fabric, and thereafter dyeing said fabric with a dyestuff selected from the group consisting of sulfur dyestuff and vat dyestuff in a conventional manner.

2. A multicolor dyed cellulose textile fabric produced according to the method defined in claim 1.

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