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SALT RINSE

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This invention relates to the rapid solution dyeing of textile materials of cellulose organic acid esters of low free hydroxyl content.

Textile materials of cellulose organic acid esters of low free hydroxyl content have many desirable properties. Thus, after such materials have been properly heat treated they may be ironed safely at high temperatures, they are resistant to glazing and to shrinkage on pressing with moist steam, and they have excellent crease-resistance, dimensional stability and washfastness. These materials are generally dyed with disperse cellulose acetate dyestuffs. However, in order to obtain washfast, fade-resistant colored materials it has been necessary to employ those disperse cellulose acetate dyestuffs which are of the "slow dyeing high temperature type." Suitable dyestuffs of this type are expensive and rather difficult to apply, particularly in the amounts necessary to produce full shades.

It is therefore an object of this invention to provide a method for dyeing cellulose organic acid esters of low free hydroxyl content easily and rapidly.

Another object of this invention is the provision of a process for the continuous dyeing of cellulose acetate of high acetyl content to produce a deeply colored material having excellent washfastness and resistance to fading.

Other objects of this invention will be apparent from the following detailed description and claims. In this description and claims all proportions are by weight unless otherwise indicated.

In accordance with this invention a textile material of fibers of cellulose organic acid esters of low free hydroxyl content is dyed for a short time in a special dyebath, identified more fully hereinafter, and without drying the wet dyed textile material the dye is fixed thereon by application of a hot salt solution thereto, whereby the textile material can be directely scoured without appreciable reduction in shade. By way of comparison, if the textile material while still wet with dyebath is rinsed and/or scoured without the salt treatment there will be an appreciable reduction in the depth of shade. While drying of the wet material prior to scouring will fix the color, such procedure will be more time consuming, will not lend itself quite as readily to continuous operation and will necessitate the expenditure of considerable additional energy to dry the material.

The process of this invention makes it possible to dye textile materials of cellulose organic acid esters of low free hydroxyl content in full shades which have excellent fastness to washing, fastness to light, and fastness to atmospheric fading agents such as ozone and nitrous oxide, and excellent resistance to crocking. In fact, textile fabrics dyed in accordance with this invention are so washfast that they pass the exceptionally severe #4 A.A.T.C.C. washfastness test, even when the fabrics have not received a subsequent heat-treatment.

As used in this application the terms "cellulose organic acid esters of low free hydroxyl content" and "highly esterified cellulose" refer to cellulose esters having less than 0.29 free hydroxyl groups per anhydroglucose unit, e.g., cellulose acetate having an acetyl value of at least 60% and preferably at least 61% calculated as combined acetic acid, hereinafter called cellulose triacetate.

The dyebaths which may be used in the practice of the invention comprise a mixture containing an organic solvent and having such solvent power that when a textile material consisting of fibers of the usual secondary cellulose acetate of lower acetyl content, having an acetyl value of 54–55% calculated as combined acetic acid, is immersed in said dye-bath, said secondary cellulose acetate fibers are coalesced and the textile material thereof loses its fibrous character, the solvent power of said mixture being, however, insufficient to cause any coalescene of or other substantial damage to the fibers of cellulose acetate of high acetyl content.

In a preferred form of the invention the dyebath contains the dyestuff dissolved in a mixture of water, an organic solvent, and a thiocyanate selected from the group consisting of alkali metal and ammonium thiocyanates.

In accordance with one particularly advantageous aspect of this invention the dyebath contains a dyestuff dissolved in a mixture of 5 to 25 parts acetic acid, 5 to 55 parts water and 25 to 70 parts of a monohydric aliphatic alcohol of the formula ROH where R is hydrocarbon, together with a thiocyanate selected from the group consisting of alkali metal and ammonium thiocyanates, the proportion of said thiocyanate in the dyebath being in the range of 5 to 12 parts and being such that when a textile material consisting of fibers of the usual secondary cellulose acetate of acetyl value 54–55%, calculated as combined acetic acid, is immersed in said dyebath said secondary cellulose acetate fibers are coalesced and the textile material thereof loses its fibrous character.

In accordance with another specific aspect of this invention the dyebath contains a dyestuff dissolved in a mixture of 5 to 35 parts water and 65 to 95 parts of a monohydric aliphatic alcohol of the formula ROH where R is hydrocarbon, together with a thiocyanate selected from the group consisting of alkali metal and ammonium thiocyanates, the proportion of said thiocyanate in the dyebath being in the range of 8 to 14 parts and being such that when a textile material consisting of fibers of the usual secondary cellulose acetate of acetyl value 54–55%, calculated as combined acid, is immersed in said dyebath, said secondary cellulose acetate fibers are coalesced and the textile material thereof loses its fibrous character.

In a further specific aspect, the dyebath liquor is a single phase solvent comprising at least 20% by weight of water and at least 30% by weight of an organic liquid swelling agent for highly acetylated cellulose, at least 10% by weight of the solvent comprising a primary swelling agent such as a polyhydric alcohol ether, a polyhydric alcohol ester, diacetone alcohol or an alkyl phosphate. These latter substances can be present to make up the full minimum of 30% swelling agent or the balance can be made up of secondary swelling agents such as carboxylic acids, e.g., formic, acetic or propionic acid, glycols, e.g., ethylene glycol, alcohols of the formula ROH, e.g., methyl, propyl, isopropyl, butyl and isobutyl alcohols and particularly ethyl alcohol, and the like. To the single phase solvent there is added an alkali metal or ammonium thiocyanate in an amount ranging from about 4 to 12% by weight of the solvent, preferably from 5 to 11%. To complete the dyebath there is dissolved in the solvent a dyestuff for the cellulose ester. The dyestuff is generally employed in an amount up to about 5% by weight of the solvent, and preferably in an amount ranging from about 0.2 to 3%. Representative primary swelling agents which can be used include β-phenoxyethanol, β-n-butoxyethanol, β-methoxyethanol, n-butoxyethoxyethanol, ethylene glycol monoacetate, glycerine diacetate, glycerine triacetate, diacetone alcohol, triethyl phosphate, tripropyl phosphate, and the like. In some instances, such as when using β-phenoxyethanol, the primary swelling agent will not be sufficiently soluble in the water so that in order to establish a single solvent phase there can be added another primary swelling agent such as diacetone alcohol or a secondary swelling agent such as acetic acid which is

miscible with both the water and β-phenoxyethanol. The use of a solvent comprising at least 20% by weight of acetic acid prevents phase separation and yields especially good dyeings, even with relatively water-insoluble primary swelling agents.

A wide variety of dyestuffs may be employed in the practice of this invention, including acid dyestuffs, premetallized dyestuffs, direct cotton dyestuffs, and leuco vat ester dyestuffs, none of which has any appreciable affinity for the cellulose acetate of high acetyl value when applied from dispersions in water containing dispersing agents. Disperse cellulose acetate dyestuffs. particularly of the known slow dyeing high temperature type may also be used. Examples of materials which have been found to be suitable include those sold under the following names.

Premetallized dyestuffs:
- Irgalan Yellow 2 RL, C.I. Acid Orange 85
- Irgalan Rubine RL, C.I. Acid Red 218
- Irgalan Bordeaux 2 BL, C.I. Acid Brown 45
- Irgalan Brown 2 RL
- Irgalan Brown 7RL
- Irgalan Orange RL, C.I. Orange 86
- Irgalan Red 3G, C.I. Acid Red 220
- Irgalan Brown 5R, C.I. Acid Brown 48
- Irgalan Brown 2GL
- Irgalan Brown, 3BL, C.I. Acid Brown 46
- Irgalan Yellow GL, C.I. Acid Yellow 114
- Irgalan Green 3GL
- Irgalan Olive BGL
- Gycolan Orange RL
- Gycolan Black WAL, C.I. Acid Black 52
- Cibalan Corinth BL
- Cibalan Green GL, C.I. Acid Green 43
- Vitrolan Orange R

Direct cotton dyestuffs: Amanil Chrysophenine G, C.I. Direct Yellow 12

Acid dyestuffs:
- Xylene Milling Yellow P, C.I. Acid Yellow 61
- Orasol Navy Blue RB
- Orasol Violet 3B
- Erio Anthracene Rubine 3GP
- Artol Blue GL (No. 833, First Edition of Color Index)
- Artol Yellow SH
- Artol Golden Brown NL
- Artol 6G
- Cloth Fast Orange G

Leuco vat ester dyestuffs:
- Indigosol Orange HR, C.I. Solubilized Vat Orange
- Indigosol Brown IRRD
- Indigosol Bordeaux 12RN
- Indigosol Green 1GG C.I. Solubilized Vat Green 2
- Indigosol Yellow R, C.I. Solubilized Vat Yellow 6
- Indigosol Red 12B
- Indigosol Olive Green 1B, C.I. Solubilized Vat Green 3
- Indigosol Brilliant Pink 15B, C.I. Solubilized Vat Red 9
- Iidigosol 04B, C.I. Solubilized Vat Blue 5
- Indigosol Scarlet RB
- Indigosol Golden Orange 12R, C.I. Solubilized Vat Orange 2
- Algosol Blue 06B, C.I. Solubilized Vat Blue 9

Disperse cellulose acetate dyestuffs:
- Lenra Blue RLS
- Lenra Yellow R
- Eastone Red NHLF
- Amacel Yellow CW, C.I. Disperse Yellow 37

Unclassified dyestuffs: Alcian Blue 6 GN, C.I. Ingrain Blue I

(C.I.—Color Index, Second Edition, 1956.)

The dyebath is preferably applied to the textile material by a padding operation in which the textile material passes through the dyebath in a pad box and then is squeezed between pad rolls to eliminate excess dyebath. The temperature at which the dyebath is applied may range from 10 to 90° C., a temperature of about 40 to 75° C. being especially suitable. With the higher temperatures the water content of the dyebath is preferably rather high and the thiocyanate content lower in order to avoid the possibility of damage due to coalescence of the cellulose ester fibers. The time of contact between the dyebath and textile material is desirably rather short, e.g., less than fifteen minutes, and preferably about 2 to 25 seconds.

Following immersion in the dyebath the textile material may be rinsed in cold water to terminate the dyeing action and to remove any loosely adherent dyestuff. The textile material wet with dyebath, or with cold water in the event that a cold rinse has been employed, is then contacted with a hot aqueous solution of a salt which dissolves to an appreciable extent in water and is highly ionized. The preferred salts are water-soluble salts of inorganic cations such as sodium, potassium, lithium, ammonium, calcium, barium, aluminum, magnesium, iron, and the like, and anions of mineral or lower aliphatic acids such as chloride, sulphate, nitrate, phosphate, bromide, fluoride, acetate, propionate, and the like, e.g., sodium sulphate, sodium nitrate, sodium acetate, sodium phosphate, potassium chloride, potassium propionate, lithium nitrate, ammonium chloride, ammonium sulphate, and the like. The best results are achieved with salts of monovalent cations such as the alkali metal and ammonium ions and the anions of mineral acids or lower alkanoic acids. Mixtures of salts can also be employed.

The salt solution should contain at least about 15 grams per liter of salt in water although small amounts of organic solvents can be included, if desired. Preferably, the solution contains at least 20 grams of salt per liter and up to about 50 grams per liter; while higher concentrations of salt are permissible they are not necessary.

The salt solution should be hot, i.e., at a temperature of at least 80° C. and preferably at least 90° C. While superatmospheric pressure can be applied to permit use of temperatures above the normal boiling points of the salt solutions, it is unnecessary. The duration of the salt solution treatment should be at least about 1½ minutes and preferably from about 2 to 10 minutes. Longer treatment times are permissible but are unnecessary.

The amount of salt solution should be sufficient fully to wet the textile material. Upon leaving the salt solution the textile material can be squeezed to expel most of the salt solution which can be re-used to minimize the amount of salt needed.

As noted previously, the textile material when wet with dyebath can be rinsed with cold water prior to the hot salt solution treatment. This is particularly desirable when the textile material contains fibers of a polyamide such as wool, Vicara (zein), silk, nylon or the like, in addition to fibers of highly acetylated cellulose and it is desired to color the highly acetylated cellulose selectively. On the other hand, if union dyeings are desired, i.e., if it is desired to color both the highly acetylated cellulose and the polyamide, there should not be a cold water rinse but rather the textile material should be passed directly from the dyebath to hot salt solution. When such a union dyeing is desired best results are achieved when the dyestuff is a premetallized or acid dye, when the salt is ammonium acetate and when the time of contact between the textile material and the hot salt solution is at least about 5 minutes. Up to about 1 gram per liter of a lower aliphatic acid such as acetic acid can be included in the hot salt solution to aid in the dyeing of the polyamide.

When the dyestuff is a leuco vat ester a cold water rinse preferably precedes the hot salt solution treatment, a suitable developing agent being included in the rinse water to develop the color; sulfuric acid in 3% concentration in the rinse water is suitable for this purpose.

Following the treatment with the hot salt solution the

textile material may be rinsed and is then scoured and dried.

The entire operation of padding and treatment with hot salt solution, plus whatever rinses are involved, may be carried out on a continuous dyeing range in which a fabric to be dyed is passed continuously at a rapid rate through a padding unit and then through a plurality of Williams Units or wash boxes of conventional construction.

While it is preferred to apply the dyebath by padding when the textile material is a fabric, other procedures are more suitable for the dyeing of other types of textile material. Thus, when a yarn is to be dyed, a skein of the yarn may be immersed in the dyebath, or the dyebath may be forced through a package of the yarn wound on a perforated core, all in a manner well known in the art of dyeing. The dyebath employed in the process of this invention may also be applied to staple fibers, as by conventional stock-dyeing techniques.

After drying following the hot salt solution treatment, the dyed textile material is preferably heat treated to increase the crystallinity and crystalline order index of the material and to raise its safe ironing point. This heat-treatment may be carried out for example in the manner described in the copending application of Salvin et al., Serial No. 472,758, filed December 2, 1954, now U.S. Patent No. 2,982,597, and Schoeneberg et al., Serial No. 565,310, filed February 14, 1956, now U.S. Patent No. 2,892,668. Such treatment improves the resistance to glazing of the dyed material and also its crease resistance and dimensional stability, and deepens the shade of the material. Before or after the heat-treatment, the textile material may be treated with any desired finishing agents such as those described in the aforementioned copending application of Salvin et al.

The fibers of cellulose acetate of high acetyl content in the textile material subjected to dyeing should not have a high degree of crystalline order such as is produced by heat treatment; the crystalline condition of the cellulose acetate fibers should be such that a fabric composed of only such fibers would have a safe ironing temperature below about 200° C.

As noted hereinabove, the textile materials can include fibers composed of substances other than highly esterified cellulose. Except when treated specially to effect union dyeings, such other fibers are either unaffected by the dyebath or are dyed by said bath only to a small extent, i.e., the depth of shade obtained on such fibers does not even approach that obtained on the fibers of highly esterified cellulose so that a cross-dyed effect is obtained. In addition to the polyamide identified hereinabove, other fibers such as polyethylene terephthalate and other polyesters, acrylonitrile polymers and copolymers, rayon, cotton, and the like, may be present in blends which are not deleteriously affected by the dyebath.

This application is in part a continuation of our copending application Serial No. 612,057, filed September 25, 1956, and Serial No. 673,167, filed July 22, 1957, both now abandoned, and the disclosures of these applications are incorporated herein by reference.

The following examples are given to illustrate this invention further.

Example I

A fabric woven of yarn of cellulose acetate of acetyl value 61.4% is padded at the rate of 35 yards per minute in a dyebath having a temperature of 32° C. and containing 45 parts of denatured ethyl alcohol, 30 parts of aqueous acetic acid of 56% concentration, 25 parts of water, 8 parts of sodium thiocyanate, 19 grams per liter of Cibalan Black BGL–200% and 13 grams per liter of Brilliant Blue Conc. The fabric, after being in contact with the dyebath for 11 seconds, is rinsed in water containing 30 grams per liter of sodium chloride at 98° C. for 2 minutes and then soaped in an aqueous solution containing 1 gram per liter of Igepon T Gel (the sodium salt of N-methyl-oleyl taurine) at 70° C. for 5 minutes. The fabric is rinsed first in hot water, then in cold water, it is extracted and dried. The fabric is colored a deep navy shade which has excellent washfastness and lightfastness. If the sodium chloride is left out of the rinse, serious reduction in depth of shade occurs during the soaping.

Example II

The process of Example I is repeated replacing the dyestuffs there disclosed with 19 grams per liter of dyebath of Glycolan Black WAL (C.I. Acid Black 52) and 13 grams per liter of dyebath of Brilliant Wool Blue GL Supra (C.I. Acid Blue 102). A similar deep navy shade results.

Example III

A fabric woven of 25/2 spun cellulose triacetate (acetyl value 61.4%) and wool in equal weights is padded at the rate of 24 yards per minute in a dyebath having a temperature of 32° C. and containing 45 parts of ethyl alcohol, 30 parts of acetic acid of 56% concentration in water, 25 parts of water, 8 parts of sodium thiocyanate, 13 grams per liter of Cibalan Black BGL 200% and 9 grams per liter of Brilliant Wool Blue GL Supra. After 11 seconds' contact with the dye liquor, the fabric passes into a 30 grams per liter solution of ammonium acetate in water and is held there for 5 minutes at 98° C. It is then rinsed with water at 25° C., soaped in 1 gram per liter of Igepon T Gel for 5 minutes at 70° C., again rinsed and dried. Both the wool and the cellulose triacetate are dyed a deep navy blue shade of excellent fastness to light and washing. If the ammonium acetate is left out of the first rinse, serious reduction in the depth of shade on both fibers occurs in the soaping. If the fabric is first run into cold water after padding and is then rinsed in salt solution before soaping, the cellulose triacetate is dyed the same navy shade, but the wool is only very lightly stained.

Example IV

The process of Example I is repeated substituting for the dyebath there disclosed a dyebath containing 30 parts of β-phenoxyethanol, 45 parts of diacetone alcohol, 25 parts of water, 9 parts of sodium thiocyanate, 19 grams per liter of Cibalan Black BGL–200% and 13 grams per liter of Brilliant Blue GL Conc.

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. A process for the coloring of textile material of a cellulose organic acid ester having less than about 0.29 free hydroxyl groups per anhydroglucose unit, which comprises applying to said textile material a dyebath containing a dyestuff dissolved in a mixture comprising an organic solvent, said mixture having such solvent power that when a textile material consisting of fibers of the usual secondary cellulose acetate of lower acetyl content, having an acetyl value of 54–55%, calculated as combined acetic acid, is immersed in said dyebath, said secondary cellulose acetate fibers are coalesced and the textile material thereof loses its fibrous character, the solvent power of said mixture being insufficient to cause any coalescence of or other damage to the fibers of the highly esterified cellulose, without drying applying to said textile material a hot aqueous solution of a salt of an acid selected from the group consisting of mineral acids and lower alkanoic acids, and subsequently scouring said textile material.

2. The process set forth in claim 1, wherein the cellulose organic acid ester is cellulose acetate having an acetyl value of at least 60%.

3. The process set forth in claim 1, wherein the dura-

tion of contact between said textile material and said dyebath is a maximum of about 15 minutes.

4. The process set forth in claim 1, wherein said salt includes a monovalent inorganic cation.

5. The process set forth in claim 1, wherein said dyebath in addition to said dyestuff comprises a solution of 5 to 25 parts of acetic acid, 5 to 55 parts of water, 25 to 70 parts of a monohydric aliphatic alcohol of the formula ROH where R is hydrocarbon, and 5 to 12 parts of a thiocyanate selected from the group consisting of alkali metal and ammonium thiocyanates.

6. The process set forth in claim 1, wherein said dyebath in addition to said dyestuff comprises a solution of 5 to 35 parts of water, 65 to 95 parts of a monohydric aliphatic alcohol of the formula ROH where R is hydrocarbon, and 8 to 14 parts of a thiocyanate selected from the group consisting of alkali metal and ammonium thiocyanates.

7. The process set forth in claim 1, wherein said dyebath in addition to said dyestuff comprises a single phase solvent consisting of at least 20% by weight of water and at least 30% by weight of an organic liquid swelling agent for said cellulose ester, at least 10% by weight of said solvent being a primary swelling agent selected from the group consisting of polyhydric alcohol ethers, polyhydric alcohol esters, diacetone alcohol and alkyl phosphates, said solvent having dissolved therein from 4 to 12% by weight of a thiocyanate selected from the group consisting of alkali metal and ammonium thiocyanates.

8. The process for the coloring of a textile material of cellulose acetate having an acetyl value of at least 60% calculated as combined acetic acid, which comprises applying to said textile material a dyebath containing a dyestuff dissolved in a mixture comprising an organic solvent, said mixture having such solvent power that when a textile material consisting of fibers of the usual secondary cellulose acetate of lower acetyl content, having an acetyl value of 54–55%, calculated as combined acetic acid, is immersed in said dyebath, said secondary cellulose acetate fibers are coalesced and the textile material thereof loses its fibrous character, the solvent power of said mixture being insufficient to cause any coalescence of or other damage to the fibers of cellulose acetate of acetyl value of at least 60%, without drying applying to said textile material an aqueous solution containing at least 15 grams per liter of a salt at a temperature of at least 80° C., said salt including a monovalent inorganic cation selected from the group consisting of the alkali metal and ammonium ions and the anion of an acid selected from the group consisting of mineral acids and lower aliphatic acids, and subsequently scouring said textile material.

9. The process set forth in claim 8, wherein the duration of contact between said textile material and said dyebath ranges from about 2 to 25 seconds.

10. The process set forth in claim 8, wherein said dyed textile material is a blend of cellulose acetate fibrous material and another fibrous material and the textile material is rinsed with cold water prior to application of the salt solution.

11. The process set forth in claim 8, wherein said textile material is a blend of cellulose acetate fibrous material and another fibrous material and the textile material is contacted with the salt solution immediately after dyeing.

12. The process set forth in claim 8, wherein the duration of contact between said dyed textile material and said hot salt solution is at least about 2 minutes.

13. The process set forth in claim 8, wherein the salt comprises sodium chloride.

14. The process set forth in claim 8, wherein the salt comprises ammonium acetate.

15. The process set forth in claim 8, wherein the salt is present in its solution to the extent of at least 5 grams per liter, the temperature of the solution is at least about 90° C. and the duration of contact between the dyed textile material and the solution ranges from about 2 to 10 minutes.

16. The process for the coloring of a textile material of cellulose acetate having an acetyl value of at least 60% calculated as combined acetic acid, which comprises contacting said textile material for about 2 to 25 seconds with a dyebath containing a dyestuff dissolved in a solution comprising 5 to 25 parts of acetic acid, 5 to 55 parts of water, 25 to 70 parts of a monohydric aliphatic alcohol of the formula ROH where R is hydrocarbon, and 5 to 12 parts of a thiocyanate selected from the group consisting of alkali metal and ammonium thiocyanates, said mixture having such solvent power that when a textile material consisting of fibers of the usual secondary cellulose acetate of lower acetyl content, having an acetyl value of 54–55%, calculated as combined acetic acid, is immersed in said dyebath, said secondary cellulose acetate fibers are coalesced and the textile material thereof loses its fibrous character, the solvent power of said mixture being insufficient to cause any coalescence of or other damage to the fibers of cellulose acetate of acetyl value of at least 60%, without drying contacting said textile material for about 2 to 10 minutes with an aqueous solution containing at least 20 grams per liter of a salt at a temperature of at least 90° C., said salt including a monovalent inorganic cation selected from the group consisting of the alkali metal and ammonium ions and the anion of an acid selected from the group consisting of mineral acids and lower aliphatic acids, and subsequently scouring said textile material.

17. The process for the coloring of a textile material of cellulose acetate having an acetyl value of at least 60% calculated as combined acetic acid, which comprises contacting said textile material for about 2 to 25 seconds with a dyebath containing a dyestuff dissolved in a solution comprising 5 to 35 parts of water, 65 to 95 parts of a monohydric aliphatic alcohol of the formula ROH where R is hydrocarbon, and 8 to 14 parts of a thiocyanate selected from the group consisting of alkali metal and ammonium thiocyanates, said mixture having such solvent power that when a textile material consisting of fibers of the usual secondary cellulose acetate of lower acetyl content, having an acetyl value of 54–55%, calculated as combined acetic acid, is immersed in said dyebath, said secondary cellulose acetate fibers are coalesced and the textile material thereof loses its fibrous character, the solvent power of said mixture being insufficient to cause any coalescence of or other damage to the fibers of cellulose acetate of acetyl value of at least 60%, without drying contacting said textile material for about 2 to 10 minutes with an aqueous solution containing at least 20 grams per liter of a salt at a temperature of at least 90° C., said salt including a monovalent inorganic cation selected from the group consisting of the alkali metal and ammonium ions and the anion of an acid selected from the group consisting of mineral acids and lower aliphatic acids, and subsequently scouring said textile material.

18. The process for the coloring of a textile material of cellulose acetate having an acetyl value of at least 60% calculated as combined acetic acid, which comprises contacting said textile material for about 2 to 25 seconds with a dyebath containing a dyestuff dissolved in a mixture comprising a single phase solvent consisting of at least 20% by weight of water and at least 30% by weight of an organic liquid swelling agent for said cellulose ester, at least 10% by weight of said solvent being a primary swelling agent selected from the group consisting of polyhydric alcohol ethers, polyhydric alcohol esters, diacetone alcohol and alkyl phosphates, said solvent having dissolved therein from 4 to 12% by weight of a thiocyanate selected from the group consisting of alkali metal and ammonium thiocyanates, said mixture having such solvent power that when a textile material consisting of fibers of the usual secondary cellulose acetate of lower acetyl con-

tent, having an acetyl value of 54–55%, calculated as combined acetic acid, is immersed in said dyebath, said secondary cellulose acetate fibers are coalesced and the textile material thereof loses it fibrous character, the solvent power of said mixture being insufficient to cause any coalescence of or other damage to the fibers of cellulose acetate of acetyl value of at least 60%, without drying contacting said textile material for about 2 to 10 minutes with an aqueous solution containing at least 20 grams per liter of a salt at a temperature of at least 90° C., said salt including a monovalent inorganic cation selected from the group consisting of the alkali metal and ammonium ions and the anion of an acid selected from the group consisting of mineral acids and lower aliphatic acids, and subsequently scouring said textile material.

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