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METHOD AND COMPOSITION FOR DYE-LEVELING

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The present invention relates to dye-leveling and, more particularly, to new and useful dye-leveling compositions for the level dyeing of textile articles produced by incorporating in the dye bath poly-N-vinyl-5-methyl-2-oxazolidinone (hereinafter referred to as PVO-M).

Although the art of dyeing textiles is relatively old, and many of the problems of dyeing are now easily coped with, one of the problems always present is that of effecting a level dyeing. What with the magnitude of dyestuffs available, and new dyestuffs being developed, along with the constantly increasing number of fibers being introduced, a means for controlling, regulating or modifying the rate of dyeing and the characteristic affinity of a particular dyestuff for a particular substrate has become increasingly necessary. Uneven or unlevel dyeing problems are even more accentuated when blends of fibers are dyed.

There are many advantages that may be derived from the preparation of mixed blends of most of the synthetic textile fibers that may be prepared from fiber-forming hydrophobic linear polymeric materials intermixed with certain of the cellulosic fibers, particularly viscose rayon, which is frequently classified as an artificial fiber that has been regenerated from a natural material, and cotton, a fiber having a natural vegetable origin, or, intermixed with protein fibers, such as wool, a fiber of animal origin. As an example of a synthetic textile fiber are the synthetic textile fibers that have been prepared from hydrophobic linear polymers and copolymers of acrylonitrile, especially those acrylonitrile polymer fibers that contain at least about 85 percent by weight of acrylonitrile polymerized in the polymer molecule, which are commonly known as being and frequently referred to as acrylic fibers. Additionally, blends of two or more of the naturally occurring fibers may advantageously be provided, such as cotton and wool.

One of the benefits that may often be derived from such fiber blends is to suppress or eliminate the undesired characteristics of the synthetic fibers that may arise from their extremely hydrophobic and electrostatic nature so that the blend behaves in the more desirable manner of the cellulosic (or other natural) fibers in those particulars. As a result, yarns and fabrics constructed with or from the fiber blends are oftentimes easier to process and handle and usually provide a better hand, or feed to the touch, and more comfortable wearing characteristics than would the synthetic textile fibers when solely employed. On the other hand, such fiber blends retain to a great degree many of the extremly desirable characteristics and properties of the synthetic textile fibers, especially as regards washability, wear properties and wrinkle resistance. Furthermore, the blends are generally more economical than the synthetic fibers alone and, in many instances, may be peculiarly attractive because of unusual or specific yarn and fabric effects that may be possibilitate.

A great disadvantage with many of the fiber blends, however, lies in the difficulty that is encountered in suitably dyeing them to uniform shades of coloration, particularly when they occur in fabric constructions. Since most synthetic fibers accept dyestuffs differently, and ordinarily with less ease, than wool or cotton and viscose

rayon cellulosic fibers, it is usually troublesome to dye fiber blends satisfactorily with the same dyestuff or even in the same dye bath. A fiber blend may frequently require that complex mixtures of different dyestuffs be employed or that plural coloring treatments be utilized in order to achieve commensurate coloration of each of the fiber materials in the blend. And, in some instances, there is no available solution to the problem of uniformly dyeing yarns and fabrics from fiber blends in a satisfactory manner.

It is the chief aim and concern of this invention to provide new and useful dye-leveling compositions suitable for promoting level dyeings with a wide variety of dyestuffs upon a wide variety of fiber substrates, especially when fiber blends are to be dyed. Additional objects and advantages of the invention will be manifest in the ensuing description and illustrations.

The level dyeings brought about by this invention are accomplished by dissolving in the dye bath poly-N-vinyl-5-methyl-2-oxazolidinone (PVO-M).

PVO-M is a water-soluble polymer of recent origin having been described in U.S. Patent No. 2,919,279. It may be prepared as high polymers having molecular weights, for example, in the range from 10 to 50 thousand and higher (as determinable from Fikentscher K-values of about 10 or more to as high as 75 to 100 or so), in order to provide a wide variety of polymer material that can be advantageously employed in the present compositions. Thus, PVO-M in broad molecular weight ranges up to 100,000 to 200,000 or so is quite soluble in water and can be beneficially used in formulating the dye-leveling compositions of the herein described invention.

The amount of PVO-M necessary to use in the dye bath to effect an advantageous level dyeing will, of course, depend upon several factors. The particular dye or dyes, the amount of dye in the bath, the fiber or fiber blend to be dyed, the temperature of dyeing, and, to some extent, the molecular weight of the PVO-M will all usually have bearing on the optimum amount of PVO-M that is to be used for any certain dyeing. These factors are, however, easily determined by a skilled artisan so that the most advantageous quantities of PVO-M can be obtained to fit any particular set of conditions. Dye baths containing as much as 20 percent of the polymer, or up to 50 or more weight percent of the polymeric solute, based on the weight of the resulting solutions, are obtainable and capable of being used if desired or required. Generally, however, quantities of PVO-M in the range from 0.1 to 50 percent based on the weight of the fiber (O.W.F.), are most beneficial for the majority of dyeings, and preferably, from 1 to 5 percent PVO-M (O.W.F.), is employed. Generally, it is desirable to use as little as possible of the PVO-M in order to avoid or minimize any losses of dyestuff that might tend to become permanently entrapped in the PVO-M.

If desirable, certain other agents may be added to the dye bath to assist in efficiently emulsifying or solvating the PVO-M, particularly if higher temperatures such as temperatures near the boil are used in the stripping operation. Beneficially, the glycol ethers described in copending application Serial No. 720,355, filed March 10, 1958, now U.S. Patent No. 3,054,764, or the alkyl diphenyl ether sulfonates, described in the copending application of William W. Bakke, entitled "Applicating Solutions of Poly-N-Vinyl-5-Methyl-2-Oxazolidinone," having Serial No. 51,483, filed August 20, 1960, now U.S. Patent No. 3,051,676, or compounds such as Aerosol OT (active ingredient, dioctyl sulfosuccinate) may be employed as assistants for emulsifying or solvating agents for the PVO-M.

The PVO-M dye leveling agent can successfully be incorporated in any of a wide variety of conventional dye baths adapted to dye raw stock, yarn, fabric or other forms of such diverse types of fibers as cotton, wool, rayon, or synthetic fibers of the cellulose acetate, acrylic, or polyamide types. Or, as a further modification, excellent results are obtained when PVO-M is incorporated in a bath suited to union dye a fiber blend selected from the above fiber types.

Thus, dyeing any of the above mentioned fibers can be beneficially improved, with respect to levelness of dyeing, by incorporating PVO-M in the dye bath selected for the fiber and containing the chosen dyestuff. Representative of dyestuffs that may be employed are:

Calcocid Alizarine Violet (Colour Index 61710, formerly Colour Index 1080), Sulfanthrene Red 3B (Colour Index Vat Violet 2), Amacel Scarlet GB (Colour Index Direct Red 1) Calcodur Pink 2BL (Colour Index 353, also more recently, Colour Index Direct Red 75), Naphthol ASMX (Colour Index 35527) and Fast Red TRN Salt (Colour Index Azoic Diazo Component 11), and Immedial Bordeaux G (Colour Index Sulfur Brown 12).

Other dyestuffs, by way of further illustration, that may be beneficially level dyed on fiber products utilizing the present invention include such direct cotton dyes as Chlorantine Fast Green 5BLL (Colour Index Direct Green 27), Chlorantine Fast Red 7B (Colour Index Direct Red 81), Pontamine Green GX Conc. 125 percent (Colour Index Direct Green 6), Calcomine Black EXN Conc. (Colour Index Direct Black 38), Niagara Blue NR (Colour Index Direct Blue 151) and Erie Fast Scarlet 4BA (Colour Index Direct Red 24); such acid dyes as Anthraquinone Green GN (Colour Index Acid Green 25) Sulfonine Brown 2R (Colour Index Acid Orange 51), Sulfonine Yellow 2G (Colour Index Acid Yellow 40), Xylene Milling Black 2B (Colour Index Acid Black 26A), Xylene Milling Blue FF (Colour Index Acid Blue 61), Xylene Fast Rubine 3GF PAT (Colour Index Acid Red 57), Calcocid Navy Blue R Conc. (Colour Index Acid Blue 120), Calcocid Fast Blue BL (Colour Index Fast Blue 59), Calcocid Milling Red 3R (Colour Index Acid Red 151), Alizarine Levelling Blue 2R (Colour Index Acid Blue 51), Amacid Azo Yellow G Extra (Colour Index Acid Yellow 63); such mordant-acid dyes as Alizarine Light Green GS (Colour Index Acid Green 25); such basic dyes as Brilliant Green Crystals (Colour Index Basic Green 1), and Rhodamine B Extra S (Colour Index Vat Blue 35); such vat dyestuffs as Midland Vat Blue R Powder (Colour Index Vat Blue 35), Sulfanthrene Brown G Paste (Colour Index Vat Brown 5), Sulfanthrene Blue 2B Dbl. paste (Colour Index Vat Blue 5), and Sulfanthrene Red 3B paste (Colour Index Vat Violet 2) as well as soluble vat dyestuffs; such acetate dyes as Celliton Fast Brown 3RA Extra CF (Colour Index Dispersed Orange 5), Celliton Fast Rubine BA CF (Colour Index Dispersed Red 13), Artisil Direct Red 3BP and Celanthrene Red 3BN Conc. (both Colour Index Dispersed Red 15), Celanthrene Pure Blue BRS 400 percent (Colour Index Dispersed Blue 1) and Acetamine Yellow N (Colour Index Dispersed Yellow 32); B-Naphthol-2-chloro-4-nitroaniline, an azoic dye; such sulfur dyes as Katigan Brilliant Blue GGS High Conc. (Colour Index Sulf Blue 9) and Indo Carbon CLGS (Colour Index Sulf. Blue 6).

In order to further illustrate the invention, the following examples are given in which all percentages are by weight unless otherwise specified.

EXAMPLE 1

Four skeins of Middling (coker) cotton 1 1/16 in. staple and 4 skeins of wool, standard white quality (all 8 skeins being of about equal weight) were pre-scoured in water with 1 percent on the weight of the fiber (O.W.F.) Dupanol WA, an anionic detergent, for 15 minutes at 71° C. The bath factor (i.e., weight of the bath liquor to weight of fiber) was about 30:1. The skeins were removed from the bath and rinsed in cold water.

A standard dyebath for acid dye application was made up as follows:

379 grams water
0.70 ml. of 28 percent acetic acid
3.8 grams Glauber's salt ($Na_2SO_4 \cdot 10H_2O$)
0.38 grams Acid Orange XX (Colour Index Acid Orange 7)

One skein of the pre-scoured cotton and one skein of pre-scoured wool, about 13.9 grams total fibers, were added to the cold dye bath, which was then heated to the boil and maintained there for 1 hour. The fibers were removed, rinsed cold, and dried.

Another dyebath was made up the same as above again using Acid Orange XX but with the addition of 1 percent PVO-M (O.W.F.) having a K-value of 30 and 0.07 gram Aerosol OT. A skein of cotton and a skein of wool were dyed as in Example 1.

The skeins dyed in the standard dyebath were observed to have light and dark regions while those dye in the presence of PVO-M were comparatively much more uniform and level in coloration.

EXAMPLE 2

According to the procedure of Example 1, a standard dyebath was formulated as follows:

420 grams water
0.7 gram 28 percent acetic acid
2.8 grams Glauber's salt
0.28 gram Wool Fast Blue BLA (Colour Index Acid Blue 59)

One skein of the pre-scoured cotton and one skein of the pre-scoured wool, about 14 grams total fibers, were added to the dye bath which had been heated to 49° C. The temperature was then raised over a 15 minute period to the boil and kept there for 45 minutes. The skeins were removed from the bath, rinsed cold, and dried.

Another dyebath was made up the same as that using the wool Fast Blue BLA but with the addition of 10 percent PVO-M (O.W.F.) having a K-value of 30, and 5 percent Aerosol OT (O.W.F.). A skein of cotton and a skein of wool were dyed according to the same procedure.

The skeins dyed in the presence of PVO-M were dyed to more level and uniform shades than those dyed in the standard dyebath.

Examples 1 and 2, then, show dyeings with standard dyeing procedures and dyeings with PVO-M added to the dye baths. From visual observation it was readily apparent that not only were the dyeings on the individlal skeins dyed in the presence of PVO-M more uniform and level, but there was a definite increase towards uniformity of shade between the cotton and wool dyed in the baths containing PVO-M.

In order to better reveal the latter mentioned aspect, numerical values for the color differences for the samples dyed with Acid Orange XX were obtained by measuring in a "Color-Eye" reflectometer. This instrument gives the so-called tristimulus color factors X, Y, and Z by comparing the colors optically with a standard white. A daylight lamp was used for illumination. To measure an X-factor, for example, the color filter X is used. What is measured then, is the ratio of the light reflectance of the sample over the reflectance of the standard white, both measured through color filter X and observed with a photoelectrical tube. A higher value means high reflectance or lighter color (closer to white). A difference of 3–5 units or so in the 20–80 percent range is considered a significant and visually observable change. The results are given in Table I.

Table I

PERCENT REFLECTANCE FOR X, Y, Z FILTERS

	X	Y	Z
Acid Orange XX:			
wool	45	20	2.5
cotton	86	69	40
wool+PVO-M	49	23	2.5
cotton+PVO-M	83	66	37

EXAMPLE 5

Samples of cotton were dyed with two different direct dyes. In each case the dyeing was performed in the presence of and in the absence of PVO-M. One set of dyeings was done with Calcomine Fast Scarlet YA (Colour Index Direct Orange 26), the other set with Calcomine Fast Black F (Colour Index Direct Black 9). The dyestuff was placed in a water bath at 160° F. The bath factor was 30:1. The cotton sample, which had been pre-scoured with 1 percent Duponal WA for 15 minutes at 160° F., was then entered and the temperature of the bath was raised to the boil over a period of 15 minutes. Twenty percent (O.W.F.) Glauber's salt crystals was slowly added to the bath and the dyeing continued for 30 minutes at which time the cotton sample was removed and rinsed in cold water, and subsequently scoured as in the pre-scour, rinsed, and dried. In the two dyeings where PVO-M was added, 1 percent PVO-M (O.W.F.) and 0.5 percent Aerosol OT were added with the dyestuff.

In both instances, the samples dyed in the presence of PVO-M were dyed to excellent level shades much superior to the samples dyed without the addition of PVO-M.

EXAMPLE 6

Samples of cotton were dyed with three different vat dyes. A dyeing was made with each dyestuff with and without PVO-M. The dyestuffs used were Calcosol Navy Blue Dbl. Pwd. (Colour Index 59810), Calcosol Jade Green NP Dbl. Pst. (Colour Index Vat Green 1), and Calcoloid Yellow GCD Dbl. Pwd. (Colour Index Vat Yellow 2). The Calcosol Navy Blue dyeing was carried out as follows: The dyestuff, 12 percent (O.W.F.) was reduced at 140° F. in a solution containing

1 gal. of H_2O per lb. of dyestuff
4 oz. caustic per gal. of H_2O
3 oz. sodium hydrosulfite per gal. of H_2O

The reduced dyestuff was then added to the dyebath (140° F.) which contained

8 lbs. of caustic per 100 gal. of H_2O
4 lbs. of sodium hydrosulfite per 100 gal. of H_2O

The cotton was then entered into the bath, the bath factor was 30:1, and dyed for a period of 30 minutes with the temperature remaining at 140° F. The cotton was then removed, rinsed lightly in cold water and placed in an oxidizing bath which contained

1.5 percent sodium dichromate
1.0 percent glacial acetic acid

The sample was oxidized for a period of 30 minutes at 160° F., then removed, rinsed well and scoured. For dyeing with the addition of PVO-M, 1 percent of PVO-M and 0.5 percent Aerosol OT were added to the dyebath immediately before adding the reduced dyestuff.

The dyeings with Calcosol Jade Green were done with 6 percent (O.W.F.) and the Calcoloid Yellow GCD with 3 percent (O.W.F.). The amounts of caustic and hydrosulfite were proportioned according to standard procedures. Other procedures were the same as the blue dyeing.

The levelness and uniformness of the samples dyed in the presence of PVO-M were strikingly better than those dyed by the standard procedures.

EXAMPLE 7

Samples of cellulose acetate fabric were dyed with dispersed acetate dyes, Celliton Fast Blue FFRN Extra Conc. (Colour Index Disperse Blue 3), and Eastman Fast Blue GLF (Colour Index Disperse Blue 27). Two dyeings were done with each dyestuff, one with and one without PVO-M. The cellulose acetate was entered into an aqueous bath (bath factor 30:1) at 160° F. containing 2 percent, O.W.F.) dyestuff. The temperature was then raised to the boil over a period of 15 minutes. The dyeing was continued 90 minutes at which time the sample was removed, rinsed, scoured and dried. One percent PVO-M (O.W.F.) and 0.5 percent Aerosol OT were added with the dyestuff for the PVO-M dyeings.

As before, the dyeings done in the presence of PVO-M were much improved over the standard procedures with respect to levelness and uniformity of shade.

Certain changes and modifications in the practice of the present invention can be readily entered into without departing from its intended spirit and scope. Therefore, it is to be fully understood that the invention is not to be considered as being limited or in any way restricted to or by the preferred deictic embodiment thereof which are included in the foregoing description and specification. Rather, it is to be interpreted and construed in the light of what is set forth and defined in the hereto appended claims taking into account the fact that poly-N-vinyl-4-methyl-2-oxazolidinone is generally equivalent to poly-N-vinyl-5-methyl-2-oxazolidinone and that identical results are obtained when the latter isomer is entirely or only partially replaced with the former and that both species, for present purposes, are adapted for utilization in the practice of the present invention and intended to be covered thereby.

It is also to be understood that certain copolymers of which N-vinyl-5-methyl-2-oxazolidinone (VO-M) is an essential ingredient may be employed in the practice of this invention. Illustrative of some of the monomers that may be polymerized with VO-M to form the copolymers are sodium styrene sulfonate, maleic acid salts, vinyl acetate, vinyl pyridine, acrylic acid, and other such water soluble monomers.

What is claimed is:

1. In the method of dyeing a textile article, the improvement of effecting a more level dyeing which consists essentially of dissolving in the dyebath with the dyestuff poly-N-vinyl-5-methyl-2-oxazolidinone and dyeing said textile article with said dyestuff in the presence of said poly-N-vinyl-5-methyl-2-oxazolidinone.

2. The method of claim 1 wherein the amount of said polymer in said dyebath is from 0.1 to about 50 percent, based on the weight of the textile article to be dyed.

3. The method of claim 1 wherein the amount of said polymer in said dyebath is from about 1 to about 5 percent, based on the weight of the textile article to be dyed.

4. The method of claim 1 wherein said textile article comprises wool.

5. The method of claim 1 wherein said textile article comprises cotton.

6. The method of claim 1 wherein said textile article comprises cellulose acetate.

7. The method of claim 1 wherein said textile article consists of a blend of at least two different kinds of textile fibers.

8. The method of claim 7 wherein the amount of said polymer in said dyebath is from 0.1 to about 50 percent, based on the weight of said blend to be dyed.

9. The method of claim 7 wherein the amount of said polymer in said dyebath is from about 1 to about 5 percent, based on the weight of said blend to be dyed.

10. The method of claim 7 wherein said fiber blend comprises a blend of wool and cotton.

11. A dyebath for dyeing textile articles consisting es-

sentially of an aqueous solvent, said solvent consisting essentially of water having dissolved therein a dyestuff for dyeing said textile articles and poly-N-vinyl-5-methyl-2-oxazolidinone.

12. The dyebath of claim 11 having dissolved therein from 0.1 to about 50 percent of said polymer, based on the weight of said incorporated textile article.

13. The dyebath of claim 11 having dissolved therein from about 1 to about 5 percent of said polymer, based on the weight of said incorporated textile article.

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