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DYEING OF WOOL WITH HIGH-AFFINITY COLLOIDAL DYESTUFFS AND DI-AMMONIUM PHOSPHATE

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The present invention relates to the dyeing of wool with the high affinity colloidal wool dyes.

The high affinity colloidal wool dyes, due to their high affinity for wool, produce colors on wool which have excellent fastness to washing and perspiration. However, because of this high affinity for wool, these dyestuffs tend to precipitate so rapidly on the fiber during the dyeing operation that it has been difficult in commercial practice to obtain level dyeing and even distribution and penetration of color.

The present invention has as an object the provision of dye-baths and dyeing processes for dyeing wool with the high affinity colloidal wool dyestuffs which will produce dyeings which have excellent fastness to washing and perspiration and which thoroughly penetrate the wool material and are evenly distributed therethrough.

In accordance with the invention we provide an aqueous dye-bath which is distinctly alkaline at the beginning of the treatment and remains distinctly alkaline, as the bath is heated, for a substantial proportion of the process. The distinct alkalinity of our bath in the early stages of the process assures retention of the dyestuff in the bath as a fine dispersion, inhibits fixation of the dye on the wool and promotes swelling of the wool fiber.

This swelling, while the dyestuff remains finely dispersed, facilitates entry of the dyestuff particles into the fiber with the result that the dyestuff thoroughly pentrates the wool material and becomes evenly distributed among and in the fibers before substantial dyeing beings.

With continued heating our dye-bath slowly and uniformly becomes less alkaline until, at about the midpoint of the process, it becomes acid. Thereafter, the bath becomes gradually and uniformly more and more acid. At these lower pH values, precipitation and fixation of the dye on and in the wool fibers takes place.

We have discovered that we can accomplish this control of the pH value of the dye-bath, with strikingly level dyeing of the wool, by including diammonium phosphate in the dye-bath.

The following table gives a typical example of

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the pH values of our dye-bath during heating of the bath.

Time (Minutes)	Temperature (Degrees, F.)	pH
0	65	7.85
15	140	7.83
30	160	7.85
45	185	7.78
60	212	7.30
75	212	6.97
90	212	6.53
105	213	6.46
120	212	6.20
135	212	6.03
150	212	5.78

The following examples are given as illustrations of our invention and not as limitations thereof. The percentages stated in the examples are by weight, based on the weight of the wool to be dyed. The dyestuff in each instance is mixed with a small amount of water before it is added to the bath.

Example I

Ten pieces, totaling six hundred pounds, of worsted gabardine cloth which had been previously chrome mordanted were run into a reel type kettle with a water ratio of about 20:01. To this 2% diammonium phosphate is added and the goods allowed to circulate in this medium for 10 minutes. The following dyestuff was then added in the indicated amounts:

Alizarine Grey 2BLW, 0.22%, Pr. 206
Omega Chrome Orange ML, .14%, Sandoz
Anthraquinone Violet 3R, .03%, C. I. 1080
Alizarine Brown BL, .40%, Sandoz
Pontachrome Grey GL, .02%, Du Pont
Polar Yellow 5G, .04%, C. I. 642

The bath was slowly brought to the boil (1½ hours) and boiling continued for 2 hours to obtain complete exhaustion. The pH at the start was about 7.1 at the boil about 6.8, and after 2 hours boil 6.5. The resulting dyeing was well penetrated, level and of good tinctorial value.

Example II

A dye-bath was prepared as in Example I and 600 pounds of similar chrome mordanted worsted gabardine cloth were dyed with the same dyeing procedure including 2% diammonium phosphate, except that after 1 hour boil 1% ammonium sulfate was added, reducing the pH to about 6.5, and after 1½ hours boiling time an additional 1% ammonium sulfate was added. The final pH was about 6.3. Chromation and exhaustion is greater by this method than by the method of Example I due to the increased acidity. The resulting dyeing was excellent for penetration, solidity and levelness of color.

Example III

Six hundred pounds of unmordanted worsted gabardine cloth comprising 10 pieces were run into a reel type kettle with a water ratio of about 20:1. To this 5% diammonium phosphate was added to also 1.0% of highly sulfonated castor oil to promote penetration and the goods were allowed to circulate in this medium for 10 minutes. 0.5% sodium bichromate dissolved in a small amount of water was then added, followed by the following dyestuff:

Alizarine Grey 2BLW, .09%, Pr. 206
Alizarine Brown BL, .075%, Sandoz
Eriochrome Flavine A, .06%, C. I. 219
Anthraquinone Violet 3R, .05%, C. I. 1080
Omega Chrome Orange ML, .033%, Sandoz

The same dyeing time and procedure was adhered to as in Example II. The resulting dyeing was excellent in penetration, solidity and levelness of color.

Example IV

A 560 lb. lot of unmordanted worsted tropical fabric was entered into a reel type kettle with a water ratio of about 20:1. To this 6% diammonium phosphate and 0.5% of a highly sulfonated castor oil were added and the goods allowed to circulate in this medium for 10 minutes. The following dissolved dyestuff was then added:

Brilliant Alizarine Blue BS, 1%, Sandoz
Brilliant Alizarine Mill, Blue BL, 1%, Sandoz
Calcocid Alizarine Blue Sky, 0.52%, C. I. 1088
Anthraquinone Violet 3R, 13%, C. I. 1080
Polar Yellow 5G, .04%, C. I. 642

The dyeing time and procedure was the same as in Example I. The resulting dyeing was excellent for penetration, solidity and levelness of color.

Example V

Forty-five pounds of worsted tops (5 lbs. each) were loaded into a Fredericks top dyeing machine with a liquor ratio of about 10:1. 4% diammonium phosphate and 1% sodium bichromate were added, followed by the following dyestuff combination:

Eriochrome Brown 3GL, .39%, Geigy
Metomega Chrome Brown RRL, .10%, Sandoz
Alizarine Grey 2BLW, .01%, Pr. 206
Pontachrome Yellow R, .01%, C. I. 343
Anthraquinone Violet 3R, .01%, C. I. 1080

The flow of liquor was from inside to outside. The dye-bath was raised to the boil in 1 hour, boiled ½ hour and exhausted by the addition of 4 to 6% of acetic acid of 56% concentration. The dyeing was excellent, with uniform color absorption from outside to center.

Example VI

Forty-five pounds of worsted tops (5 lbs. each) were loaded into a Fredericks top dyeing machine with a liquor ratio of about 10:1. 5% diammonium phosphate was added, followed by the following dyestuff combination.

Eriochrome Brown 3GL, .39%, Geigy
Metomega Chrome Brown RRL, .10%, Sandoz
Alizarine Grey 2BLW, .01%, Pr. 206
Pontachrome Yellow R, .01%, C. I. 343
Anthraquinone Violet 3R, .01%, C. I. 1080

The flow of liquor was from the inside to the outside. The dye-bath was raised to the boil in 1 hour, boiled ½ hour and exhausted with 2% of acetic acid of 56% concentration. 1% of sodium bichromate was added to the bath after exhaustion and boiling continued for 1 hour. The dyeing was excellent, with uniform color absorption from outside to center.

Our process is subject to variation of the conditions described and illustrated in the examples. The process is applicable to wool in any form, such as raw stock, top, slubbing, roving, and woolen or worsted yarn or fabric, and is applicable to the chrome mordant, metachrome or top chrome dyeing processes and to processes in which no mordant is employed.

The process also is applicable to all dyestuffs except the acid dyes, and may be used, for example, with dyes of the following classes.

I. Chromate or metachrome dyes, such as:

a Chrome Brown RLL
b Monochrome Black Blue G
c. Chrome Brown 3GL

II. Neutral or Milling dyes, such as:

a. Polar Red G
b. Polar Orange R
c. Alizarine Milling Blue BL
d. Alizarine Light Brown BL

We refer herein to the dyestuffs to which the invention is applicable as the high affinity colloidal wool dyes.

While we prefer to enter the wool into the dye-bath at room temperatures, as is illustrated in the examples, our process is effective if the wool is entered at any temperature below the boil.

The amount of diammonium phosphate to be included in the dye-bath may vary from about 0.1% to 0.5% of the total weight of the bath, which will usually be about 2% to 6% of diammonium phosphate based on the weight of the wool. The amount to be used, within this range, can readily be determined by the textile dyer.

The process of the present invention provides dyeings of excellent penetration, levelness and solidity of color on woolen or worsted piece goods, which heretofore have not been completely satisfactorily dyed with the high affinity colloidal dyes by the existing commercial methods.

We claim:

1. The process for the dyeing of wool with the high affinity colloidal wool dyes which comprises forming a distinctly alkaline dye-bath by adding to water a high affinity colloidal wool dye and an amount of diammonium phosphate between about 0.1% and 0.5% of the weight of the bath and between about 2% and 6% of the weight of the wool to be dyed, wherein the high affinity colloidal wool dye is present as a dispersion, immersing the wool in said dye-bath at room temperature, heating the bath slowly below its boiling temperature for about one to one and one

half hours while maintaining the pH of the bath distinctly alkaline, retaining dyestuff in dispersed form in the bath and inhibiting dyestuff fixation on the wool, whereby the wool fiber is swollen and the dyestuff particles are evenly distributed therethrough, and then bringing the bath to the boil and maintaining it at the boil as it becomes distinctly acid by the liberation of ammonia, thus causing fixation of the dyestuff particles on the wool evenly distributed therethrough.

2. The process for the dyeing of chrome mordanted wool with the high affinity colloidal wool dyes which comprises forming a distinctly alkaline dye-bath by adding to water a mordantable high affinity colloidal wool dye and an amount of diammonium phosphate between about 0.1% and 0.5% of the weight of the bath and between about 2% and 6% of the weight of the wool to be dyed, wherein the high affinity colloidal wool dye is present as a dispersion, immersing the wool in said dye-bath at room temperature, heating the bath slowly below its boiling temperature for about one to one and one half hours while maintaining the pH of the bath distinctly alkaline, retaining dyestuff in dispersed form in the bath and inhibiting dyestuff fixation on the wool, whereby the wool fiber is swollen and the dyestuff particles are evenly distributed therethrough, and then bringing the bath to the boil and maintaining it at the boil as it becomes distinctly acid by the liberation of ammonia, thus causing fixation of the dyestuff particles on the wool evenly distributed therethrough.

3. The process for the dyeing of wool with the high affinity colloidal wool dyes which comprises forming a distinctly alkaline dye-bath by adding to water a mordantable high affinity colloidal wool dye, a chrome mordant and an amount of diammonium phosphate between about 0.1% and 0.5% of the weight of the bath and between about 2% and 6% of the weight of the wool to be dyed, wherein the high affinity colloidal wool dye is present as a dispersion, immersing the wool in said dye-bath at room temperature, heating the bath slowly below its boiling temperature for about one to one and one half hours while maintaining the pH of the bath distinctly alkaline, retaining dyestuff in dispersed form in the bath and inhibiting dyestuff fixation on the wool, whereby the wool fiber is swollen and the dyestuff particles are evenly distributed therethrough, and then bringing the bath to the boil and maintaining it at the boil as it becomes distinctly acid by the liberation of ammonia, thus causing fixation of the dyestuff particles on the wool evenly distributed therethrough.

4. The process for the dyeing of wool with the high affinity colloidal wool dyes which comprises forming a distinctly alkaline dye-bath by adding to water a mordantable high affinity colloidal wool dye and an amount of diammonium phosphate between about 0.1% and 0.5% of the weight of the bath and between about 2% and 6% of the weight of the wool to be dyed, wherein the high affinity colloidal wool dye is present as a dispersion, immersing the wool in said dye-bath at room temperature, heating the bath slowly below its boiling temperature for about one to one and one half hours while maintaining the pH of the bath distinctly alkaline, retaining dyestuff in dispersed form in the bath and inhibiting dyestuff fixation on the wool, whereby the wool fiber is swollen and the dyestuff particles are evenly distributed therethrough, and then bringing the bath to the boil and maintaining it at the boil as it becomes distinctly acid by the liberation of ammonia, thus causing fixation of the dyestuff particles on the wool evenly distributed therethrough, adding a chrome mordant to the bath and thereafter further boiling the wool in the bath.

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