PROCESS FOR DYING TEXTILE FIBERS WITH VAT DYES

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This invention relates to an improvement in the process for dyeing textile fabrics with vat colors, and, more particularly, to a process whereby piece goods may be dyed in a continuous manner by a pigment pad dying method.

There are two general methods in practice today for dyeing piece goods with vat dyes. The first process, and the only one which has been adapted for the continuous dying of piece goods as distinguished from batch processes, is the one in which the vat color is applied to the goods in the reduced form. Such processes are illustrated in the patent to Tietz, U. S. P. 1,652,649, and the more recently developed process of Wentz U. S. P. 2,318,133. The dyeing of the fiber from solutions of the reduced color presents some difficulties, however. In the first place, the amount of dye that can be applied to the fiber will depend upon how soluble the color is in the reduced form, and the affinity of the dye for the fiber. Many of the anthraquinone vat colors are not readily soluble in the vat, and therefore it is difficult to apply them in the heavy shades required, without running the goods through the dye bath a second or third time. It has also been found difficult to dye cotton goods evenly in very weak or pastel shades by the reduced-pad method, because of the tendency of the color to migrate on the goods in the presence of the comparatively large amount of liquid that is taken up by the goods in the dyeing operation. It has also been found that, when dyeing in heavier shades with those vat dyes that are sufficiently soluble to be applied in heavy shades, it is difficult in many cases to obtain the desired penetration. The reduced vat dying processes also require large volumes of liquid from which the dye must be applied, for even with the more soluble types they have limited solubility even in reduced form.

The second method generally employed in the dyeing of piece goods in what is commonly referred to as the pigment-pad reduction dying process, or the "pad-jig" process. In this process, the dye in a highly dispersed (unreduced) form is applied to the fiber from concentrated suspensions, and this finely suspended dye is forced into the fabric as the cloth passes between squeeze rolls. The cloth is then run into rolls either without or after drying. These rolls are then put in a jig, in which the goods is run back and forth several times through an alkaline reducing solution until the reduction and fixation of the dye on the fiber is complete. The goods is then run through an oxidizing bath or otherwise oxidized, then rinsed, soaped, re-rinsed and dried in the customary manner. This pad-jig process permits the application of the dye in as heavy strengths as desired, and the amount of water employed can be reduced to only that required to carry the suspended color into the fiber under pressure of the squeeze rolls. This process, however, requires a large amount of handling of the goods and an unnecessary length of time in passing the goods back and forth through the reducing solution on the jig. Furthermore, because the fabric containing the reduced solution of the dye comes in contact with air, there is some surface oxidation which is objectionable.

It is therefore an object of this invention to provide a process for applying vat dyes to textile fibers by a pigment-pad process wherein the goods can be continuously padded and developed without extra handling and without any chance of surface oxidation of the color on the fiber prior to complete development of the color thereon. A still further object is to provide a continuous piece goods dyeing process which will give results equally good or better than those obtained by the known reduced dyeing processes, particularly as to penetration and scrub fastness, and at the same time will obviate the handling of goods required in the usual pad-jig method, and thereby speed up production.

It is a still further object of this invention to provide a continuous piece goods dyeing process which will require the minimum volumes of dye and chemical solutions, whereby greater output per unit may be attained, and wherein the bleed-off in the developing solution may be held to a minimum. It is a further object to provide a process for dyeing piece goods in a continuous manner which can be employed in applying colors in heavy shades in a single operation, such dyeing being equal in value to those produced in the normal pad-jig method.

I have found that textile fibers may be dyed with vat dyes by a continuous dyeing process by padding the vat color on to the cloth in pigment form, such as in the usual pigment-pad method, then passing the cloth padded with the color through a reducing solution at a temperature below that at which actual reduction of the dye takes place, and then immediately passing the goods into an air-free development chamber wherein the goods are alternately subjected to the action of steam and an alkaline reducing solution held at boiling temperature. By this process, reduction and fixation of the color on the fiber takes place rapidly, and, because the amount
of developing solution, namely, an alkali and hydrosulfitite, in the developing chamber is kept at a minimum, equilibrium between the amount of color bleeding-off of the goods into that solution, as more particularly disclosed and considered in Wentz U. S. P. 2,318,133 above-mentioned, is reached quickly.

By permitting the greatest amount of the development to be carried out in the steam chamber above the developing bath and in the absence of air, the amount of chemicals that must be continuously added to the developing solution is held to a minimum.

A more complete understanding of the process of this invention can be obtained by reference to the drawing which forms a part of this specification and which contains a diagrammatical representation of how the goods may be dyed and developed in a continuous manner by this process.

In the drawing, the numeral 1 refers to the container in which the dye padding solution is held and in which the goods 2 is padded in the usual manner with the pigment dispersion of the desired dye. It is then passed through squeeze rolls 3, by which the amount of moisture and color to which the goods 2 is control. The goods is then preferably passed through a hot flue 4, whereby it may be dried by the means of air or other gases. The dye goods, impregnated with the dye, is then passed through the chemical pad 5 containing the caustic alkali and hydrosulfitite or other reducing agent, and through squeeze rolls 6, whereby the amount of liquid to be retained in the goods can be controlled, after which it passes between guide rolls 7 into the developing chamber 8, over and under rolls 9 and 10, respectively, and then out of the developing chamber. After leaving the developing chamber, the goods containing the dye developed therein is passed through squeeze rolls 11 and then subjected to the usual oxidizing, rinsing, soaping, rinsing, and drying processes, all of which can be carried out in the continuous piece in the same manner as in the reduced-pad continuous dyeing processes.

For convenience, the developing chamber 8 is divided into sections by partitions 15 and 16, so that it is possible to employ solution in only one or two of the sections, depending upon the amount of time that the particular fiber impregnated with dye and reducing agent is to be subjected to the steam atmosphere, and for how long or how often it is to be immersed in the alkali developing solution to obtain the desired result.

Steam may be introduced through inlets 17 or 18, or both, to maintain the solution and atmosphere at the desired temperature.

Although, as indicated in the drawing, the goods after being padded with the dye is preferably passed through a hot flue or over other drying apparatus before it is impregnated with the chemical pad, this is not an essential requirement in the process. If the chemical pad is applied to the wet pigmented goods, the moisture content of the final chemical padded goods should not be over 100%, based on the weight of the dry goods. Control of this moisture content of the impregnated fabric prior to its passage into the developing chamber has been found desirable. In the production of level dyeings, for excessive moisture will tend to cause streaking. With most dyes and with most fabrics, satisfactory results are obtained with the moisture content of the goods, as it enters the developing chamber, maintained at between 50% and 100%, based on the weight of the fabric.

As illustrated in the diagrammatic sketch, suitable means are provided for introducing the developing solution into the various compartments and for maintaining any predetermined level of the solution in the compartment.

By passing the goods directly from the reducing pad into the developing chamber, from which all air or oxygen is excluded, the reduction and fixation of the color is rapidly effected. Where maximum penetration, or, in other words, minimum migration of the dye to the surface, is desired, the number of dips of the cloth into the developing solution in the developing chamber is reduced to a minimum. In certain cases, however, it is desired to increase the apparent strength of the dye on the goods by causing some small amount of migration of the dye to the surface of the fiber. By the process of this invention, wherein the development is carried out partly in the steam above the developing bath, the amounts of migration can be carefully controlled so that stronger dyeings are obtained without the migration from the center of the goods to such a degree as to detract from the usefulness of the fabric for most purposes.

To prevent a tapering of the color on the goods at the start of the dyeing, a predetermined amount of dye is added to the alkaline-reducing solution in the developing chamber, as more particularly described in the development of reduced-pad dyeings in the '156 and the Wentz patents above-mentioned. However, if no dye is added an equilibrium is soon reached, and the dyeing then remains at a constant shade.

The development of the vat dye on the fiber at temperatures of 212° F. or somewhat higher has been found to very greatly increase the speed at which the dyeing can be effected, and also has been found to improve the quality of the resulting dyeings with regard to penetration and resulting fastness, particularly as to laundring and scrubbing.

To prevent premature reduction of the dye on the fiber after passing it through the chemical-padding solution, the chemical-padding operation, namely, the application of the alkaline reducing solution, should be carried out at temperatures preferably not over 90° F. Temperatures as high as 110° F. may be employed, provided the time of immersion in the chemical solution is held at a minimum and the goods is passed immediately into the developing chamber.

The concentration of the caustic and hydrosulfitite in the chemical bath may be varied widely as long as sufficient is employed so that, for the particular moisture retention allowed, complete reduction and development in the developing chamber is effected. Where the pudder is set to retain 50% moisture from the chemical-padding operation, a concentration of 4 ounces of caustic and 4 ounces of hydrosulfitite per gallon is usually sufficient. The time of immersion of chemical pad should be from 2 to 10 seconds, although a longer time may be required for certain types of fabrics. The time of immersion should be kept at the minimum required to obtain complete penetration of the chemical solution to the goods.

Following the chemical-padding operation, it is desirable that the goods be passed directly and quickly as possible into the developing chamber to avoid any tendency toward decomposition of the reducing agent, or oxidation of any
Experience has shown that no appreciable decomposition can be detected when the padding is carried out at temperatures not higher than 90°F. and after 10 seconds exposure prior to entering the developing chamber. This permits considerable leeway in leading the goods from the chemical pad to the developing chamber.

The temperature in the developing chamber is held at, or slightly above, the boiling point of the developing bath. While higher temperatures may be employed, their use requires the use of increased pressure in the developing chamber so that the developing solution will not be vaporized. With increased temperatures above the boiling point of the liquid, a reduction in the time required to effect complete development can be attained. At temperatures of 212°F., or slightly above, substantially complete reduction and fixation of the dye is obtained in from 30 to 60 seconds. Longer periods in the developing bath may be required with certain of fiber. After development and fixation in the developing chamber, the cloth is run into the usual oxidizing baths. Chrome acid, sodium perborate, hydrogen peroxide, or other oxidizing agent may be employed, or the dye may be oxidized on the goods by means of air. After oxidation, the goods is soaked, rinsed and dried in the usual manner.

To facilitate the chemical-paddling operation, wetting agents may be employed “Alkanol” B (sodium alkyl-naphthalene sulfonate) operates very effectively for this purpose when added to the pigmented-pad bath.

The following examples are given to illustrate the invention. Unless otherwise specified, the parts used are by weight.

**Example 1**

A mixture containing 0.25 oz. per gallon of the dyestuff of Example 1 of U. S. P. 2,212,029, 0.37 oz. per gallon of pentanthrime carbazole (U.S. P. 2,028,103), and 0.44 oz. per gallon of the dyestuff of U. S. P. 1,525,117 is padded on herringbone twill with the pad set for 50% moisture retention, and the impregnated goods then dried.

The impregnated fabric is then padded a second time with 50% moisture retention with a solution consisting of 1.5 oz. per gallon of caustic soda and 4 oz. per gallon of sodium hydrosulfite at a temperature of 70°F. The fabric is then passed through the developing chamber, as illustrated in the accompanying drawing, where the developing solution is present in all three of the compartments, at such a rate that it is subjected to the action of the steam and the developing solution containing 1.5 oz. per gallon of caustic soda and 1.5 oz. per gallon of sodium hydrosulfite, for a period of one minute. The amount of solution maintained in the bottom of the developing chamber is such that, for one-fifth of the total time the cloth is in the developing chamber, it is passing through the developing solution. The liquid in the booster is under a head of steam at 212°F. The impregnated cloth is then oxidized in the conventional manner in a 0.25% chrome acetic acid solution, rinsed, soaped, rinsed and dried. The fabric is dyed an olive drab shade with good penetration and fastness to laundering and abrasion.

**Example 2**

The fabric is padded with a dyestuff suspension and in the chemical pad, as in the preceding example, and then passed in one minute through a three-section booster, as illustrated in the drawing, of which only the third section contains caustic soda and hydrosulfite in the proportions as in the preceding example, so that, while in the developing chamber, the goods is immersed in the developing solution for only one-fifteenth of the time. The goods is oxidized in the conventional manner. The dyeing is apparently somewhat weaker (5%) than that in the preceding example, but the penetration and the fastness to laundering and abrasion are somewhat better.

**Example 3**

The fabric is padded with a dyestuff suspension and the reducing solution as in Example 1, and then passed in one minute through a three-section booster, as in Example 2, where only the third section contains 1.5 oz. per gallon caustic soda, 1.5 oz. per gallon of sodium hydrosulfite and 0.10 oz. per gallon of the dyestuff mixture in the proportions given in Example 1. The impregnated cloth is then oxidized in the conventional manner in a 0.25% chrome acetic acid solution, rinsed, soaped, rinsed and dried. The fabric is an olive drab shade approximately 5% to 10% stronger than that obtained by the process of Example 1. Penetration was excellent, and the fastness to laundering and abrasion was fully equal to that of Example 2.

**Example 4**

A mixture containing 0.621 oz. per gallon of pentanthrime carbazole, 0.587 oz. per gallon of the dyestuff of Example 1 of U. S. P. 2,212,029, 0.104 oz. per gallon of the dyestuff of Color Index #1150, 0.104 oz. per gallon of the dyestuff of Color Index #1151, and 0.400 oz. per gallon of “Alkanol” B (sodium alkyl naphthalene sulfonate) is padded on herringbone twill with the pad set for 50% moisture retention, and the impregnated goods then dried.

The impregnated fabric is then padded a second time with 50% moisture retention with a solution consisting of 6 oz. per gallon of caustic soda, 6 oz. per gallon of sodium hydrosulfite and 6 oz. per gallon of common salt, at a temperature of 50°F. The fabric is then passed in one minute through a steam development chamber containing a three-section booster, as in Example 2, where only the third section contains 1.5 oz. per gallon of soda ash, 0.05 oz. per gallon of caustic soda, 0.40 oz. per gallon of sodium hydrosulfite, and 4 oz. per gallon of common salt at 212°F. Sections one and two contain only steam. The impregnated cloth is then oxidized in the conventional manner in a 0.25% chrome acetic acid solution, rinsed, soaped, rinsed, and dried. The fabric an olive-drab shade approximately 10% stronger than the jig development. Fastness to rubbing and laundering is noticeably superior to both the jig and reduced pad-continuous processes. The penetration is equal to the jig process but noticeably superior to the reduced pad-continuous processes.

Additions of 0.1% to 10% of the concentration of color in the pigment-padd may be added to the booster bath to obtain additional strength in the dyeing.

As illustrated in the above examples, the amount of time in which the goods is developed in the steam and in the developing solution can be varied by employing developing solution in only one or more of the compartments, as well
As by changing the depth of the developing solution through which the goods must travel. With certain types of fiber, it has been found desirable that the goods while in the developing chamber should be subjected several times to the developing solution, while with other types of goods and dyes only one pass into the developing solution may be required to produce the desired result.

It is preferable that the cloth on entering the developing chamber be heated quickly to the temperature of the chamber, and the heating should be carried out in such a manner that it is uniform on both sides of the fabric. The flow of steam through the apparatus should be regulated in such a manner that the decomposition products of the reducing agent, etc., are continually removed. The formation or deposition of moisture as condensate on the fabric should be avoided.

The process of this invention may be employed in the dyeing not only of cellulosic materials, but in the dyeing of other fibers and mixed fabrics, for example, those containing or consisting of nylon, viscose-cotton mixtures, acetate-rayon viscose mixtures, etc. It is of course understood that other vat dyes than those specifically employed above may be dyed by this method, such as the thio-indigos, sulfur colors, etc., which are dyed by the vat dyeing method.

In place of sodium hydroxosulfite, other reducing agents which operate in a similar manner may be employed. The time required for complete reduction and development in the developing chamber will depend upon the ease with which the particular color is reduced and upon the type of fiber being dyed. The process is particularly suitable for dyeing very lightly woven fabrics, such as Indian Head fabric, in even shades. This type of goods is difficult to dye in even shades by the ordinary reduced-pad dyeing methods.

This process provides a satisfactory method for dyeing goods in the continuous piece, where the dye is applied by pigment-pad process. Because the development is carried out partly in steam and with only a small volume of developing solution, the amount of chemicals and dye required in the developing bath is reduced to a minimum. The process permits the dyeing in a continuous manner by pigment-pad method, whereby heavy shades can be obtained with dyes that will not produce heavy dyehays by the usual reduced-pad dyeing methods. The dyehays exhibit excellent penetration and have fastness properties approximating the fastness of the dyehays obtained by the usual pad-jig dyeing method. They exhibit substantially better fastness to rubbing and laundering than the dyehays obtained by the continuous reduced-pad dyeing method.

The process of this invention is applicable for the dyeing of warps, warps, or any other form of textile fiber which can be introduced into the pigment pad, chemical pad, and development chamber in a continuous manner.

Other advantages of the pigment-pad dyeing processes heretofore operated are obtained in dyeing piece goods in the continuous manner, as distinguished from the pad-jig methods heretofore required.

I claim:

1. In a continuous process for dyeing textile fibers with vat dyes, the steps which comprise padding the fiber, which has been impregnated with unreduced dye in pigment form, with a caustic alkalai solution of a reducing agent under conditions which do not immediately reduce the vat color on the fiber, and then alternately subjecting the fiber padded with color and reducing agent to the action of steam and an alkalai solution of a reducing agent to effect development of the color on the fiber, said development treatment being continued until substantially all of the color has been reduced and fixed in the fiber, said treatment in alkalai reducing agent and steam being carried out in the absence of air.

2. In a continuous process for dyeing textile fibers with vat dyes, the steps which comprise padding the fiber, which has been impregnated with unreduced dye in pigment form, with a caustic alkalai solution of a reducing agent under conditions which do not immediately reduce the vat color on the fiber, and immediately subjecting the fiber padded with color and reducing agent to the action of steam and then alternately to the action of steam and an alkalai solution of a reducing agent to effect development of the color on the fiber, said development treatment being continued until substantially all of the color has been reduced and fixed in the fiber, said treatment in alkalai reducing agent and steam being carried out in the absence of air.

3. The process of claim 1, in which some of the color originally employed in the padding of the fiber is added to the development solution to prevent initial bleeding-off of the color in the fiber into the developing bath.

NICHOLAS R. VIEIRA.

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