Aug. 24, 1948. N. R. VIEIRA 2,447,993

PROCESS FOR DYEING TEXTILE FIBERS WITH VAT DYES

Filed Feb. 22, 1945

Fig. 1.

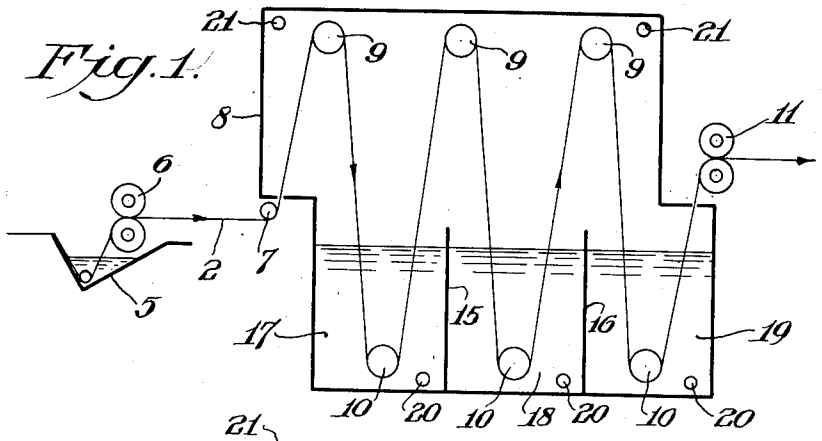


Fig. 2.

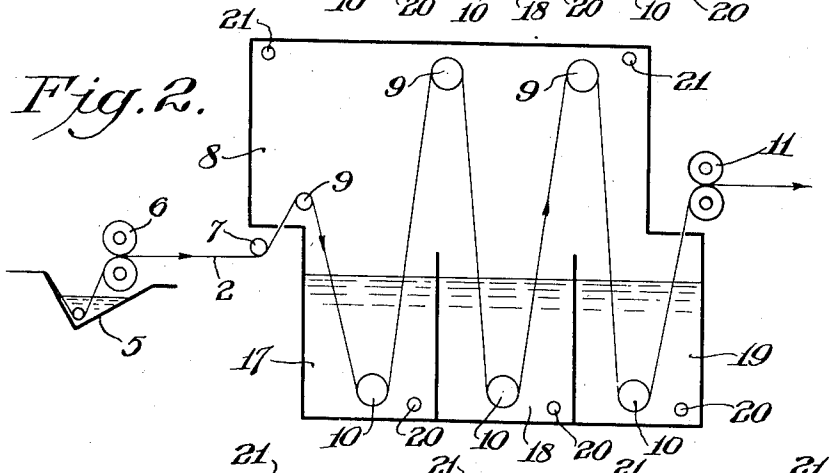
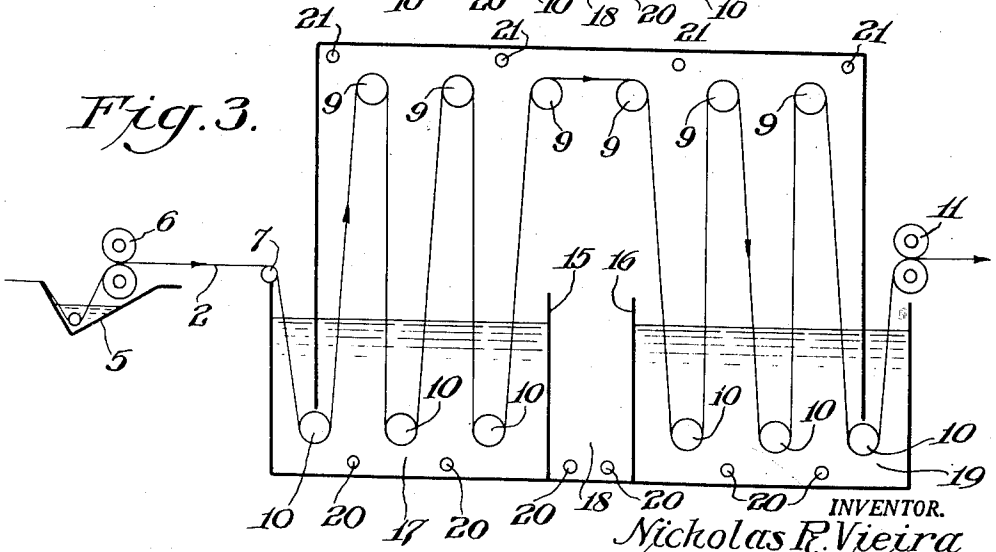


Fig. 3.



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PROCESS FOR DYEING TEXTILE FIBERS WITH VAT DYES

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2 Claims. (Cl. 8—34)

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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 dyeing method.

There are two general methods in practice today for dyeing piece goods with vat dyes. The first process 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 Tice, 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 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 dyeing 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 is what is commonly referred to as the pigment-pad reduction dyeing 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

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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 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 small 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.

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, and then immediately passing the goods into an air-free development chamber wherein the goods is alternately subjected to the action of steam and a caustic 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, caustic and hydrosulfite, in the developing chamber is kept at a minimum, equilibrium between the amount of color bleeding-off of the goods into that solution 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 continually 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, which diagrammatically illustrates how this invention may be carried out, the numeral 5 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 6, whereby the amount of liquid to be retained in the goods can be controlled, after which it passes about the 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.

The developing chamber 8 is divided into sections 17, 18 and 19 by partitions 15 and 16, so that solutions of varying strengths may be employed in the different sections, or, in cases where desired, a solution may be employed in only one section, depending upon the time that the particular fiber impregnated with dye is to be subjected to the steam atmosphere and to the developing solution. The ratio of time in the developing solution and in the steam can be varied in order to obtain any particularly desired result. In each case, however, the first section 17 must always contain a reducing solution since no previous chemical padding with a reducing solution is employed outside of the developing chamber. Steam may be introduced through inlets 20 and 21, or both, in order to maintain the solution and the atmosphere above the solution at the desired temperature. As illustrated in Figures 1 and 2 of the accompanying drawing, the goods may first be heated in the steam or immediately immersed in the reducing solution, as desired.

Suitable means may be provided for introducing the developing solution into the various compartments and for maintaining it at any predetermined level.

By passing the goods directly from the pigment pad into the developing chamber, from which all air or oxygen is excluded, the reduction and fixation of the color is rapidly effected. The reducing solution, which contains sodium hydrosulfite and a strong alkali such as caustic soda, is at the boiling temperature, and the remainder of the developing chamber is filled with an atmosphere of steam. The fabric padded with the vat color in pigment form picks up the necessary reducing agent for conversion of the color to its substantive leuco derivative during the immersion in the liquid phase in the developing chamber. Reduction and fixation of the leuco derivative is effected in both the liquid and steam phases. However, since the time of exposure to steam is greater than the time of exposure to the liquid reducing solution in the developing chamber, the major proportion of the reduction and fixation takes place in the steam phase. This is an important feature of the invention, since it includes the advantages of steam fixation as described in my copending application Serial No. 572,852 and U. S. Patent 2,415,379.

Since the leuco derivative of the vat color is soluble in the reducing solution, equilibrium is established between the vat color present on the fabric and in the reducing solution. 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 Tice and the Wentz patents abovementioned. 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 laundering and scrubbing.

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 ounce per gallon of the dyestuff of Example 1 of U. S. P. 2,212,029, 0.4 ounce per gallon of the dyestuff of U. S. P. 2,028,103 (pentanthrimide carbazole) and 0.042 ounce per gallon of the dyestuff obtained by chlorination of the material described in Example 5 of U. S. P. 1,924,456, is padded at 140° F. on cotton herringbone twill fabric with the padder set for 50% moisture retention and then passed, without drying, 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 ounces per gallon of caustic soda and 1.5 ounces per gallon of sodium hydrosulfite, for a period of two minutes. 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 compartments is held at 212° F. throughout the run. 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 as in Example 1, and then passed in two minutes through a three-section booster, as in Example 1, where only the first and second sections contain 1.5 ounces per gallon of caustic soda and 1.5 ounces per gallon of sodium hydrosulfite and 0.06 ounce per gallon of the dyestuff mixture in the proportions given in Example 1. The third section contains only steam. In this case, the fabric is passing through the developing solution for approximately one-tenth of the total time it is in the developing chamber. 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 slightly stronger in appearance 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 1.

The concentration of the caustic and alkali

metal hydrosulfite 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 padder is set to retain 50% moisture from the pigment-padding operation, a concentration of from 1.5 to 2.5 ounces per gallon each of caustic and hydrosulfite is usually sufficient.

The determination of the total time of immersion of the fabric in the reducing solution while in the development chamber is based on the nature of the fabric, i. e., its absorbency and penetrability by the reducing solution, the concentration of reducing agent, and the depth of shade being produced. These factors, of course, will vary in practice. The total time of exposure of the fabric in the development chamber likewise will vary, dependent on the same factors. Uusually, the total time of exposure is approximately 90 seconds, and in no case is more than 165 seconds required. In every instance the time of exposure to the steam phase is greater than the time of immersion in the reducing solution, but best results usually are obtained if the time of exposure to steam is at least four times the time of immersion in the reducing solution.

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.

After development and fixation in the developing chamber, the cloth is run into the usual oxidizing baths. Chromic 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 soaped, rinsed and dried in the usual manner.

To facilitate the vat dye pigment-padding operation, wetting agents may be employed. "Alkanol" B (sodium alkyl-naphthalene sulfonate) operates very effectively for this purpose when added to the pigment-pad bath.

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.

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 hydrosulfite, 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.

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 dyeings by the usual reduced-pad dyeing methods. The dyeings exhibit excellent penetration and have excellent fastness properties. They exhibit substantially better fastness to rubbing and laundering than the dyeings obtained by the continuous reduced-pad dyeing method.

The process of this invention is applicable for the dyeings of warps, skeins, or any other form of textile fiber which can be introduced into the pigment 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.

The attached drawing indicates the preferred type of apparatus for carrying out this new process on woven fabrics. However, it will be understood that this invention relates to a new dyeing process, and other apparatus in which substantially identical dyeing conditions can be produced may be utilized to advantage in some instances. For example, the new vat dyeing processes may be conducted in the multiple-lap dyeing machine described in Wentz U. S. P. 2,369,696 and in application Serial No. 577,006. Likewise, other apparatus or modifications of the apparatus illustrated in the drawing will be required in the application of the invention to the dyeing of warps, skeins, and other forms of textile fibers.

I claim:

1. In a continuous process for dyeing textile fibers with vat dyes, the steps which comprise padding the fiber with the unreduced vat dye in pigment form, immediately passing the fiber padded with the color into a closed chamber containing steam and an alkaline solution of a reducing agent maintained at boiling temperature and capable of completely reducing the vat dye, and alternately subjecting the fiber padded with color to the action of the alkaline solution of the reducing agent and to the action of the steam to effect reduction and development of the color on the fiber, the time of exposure to steam exceeding the time of immersion in the alkaline reducing solution, said development treatment being continued until substantially all of the color has been fixed in the fiber, said treatment in the alkaline treating solution 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 with the unreduced vat dye in pigment form, immediately passing the fiber padded with the color into a closed chamber containing steam and an alkaline solution of a reducing agent maintained at boiling temper-

ature and capable of completely reducing the vat dye, and alternately subjecting the fiber padded with color to the alkaline solution of the reducing agent and to the action of the steam to effect reduction and development of the color on the fiber, the time of exposure to steam being at least four times as long as the time of immersion in the alkaline reducing solution, said development treatment being continued until substantially all of the color has been fixed in the fiber, said treatment in the alkaline treating solution and steam being carried out in the absence of air.

NICHOLAS R. VIEIRA.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,712,044	Macadam	May 7, 1929