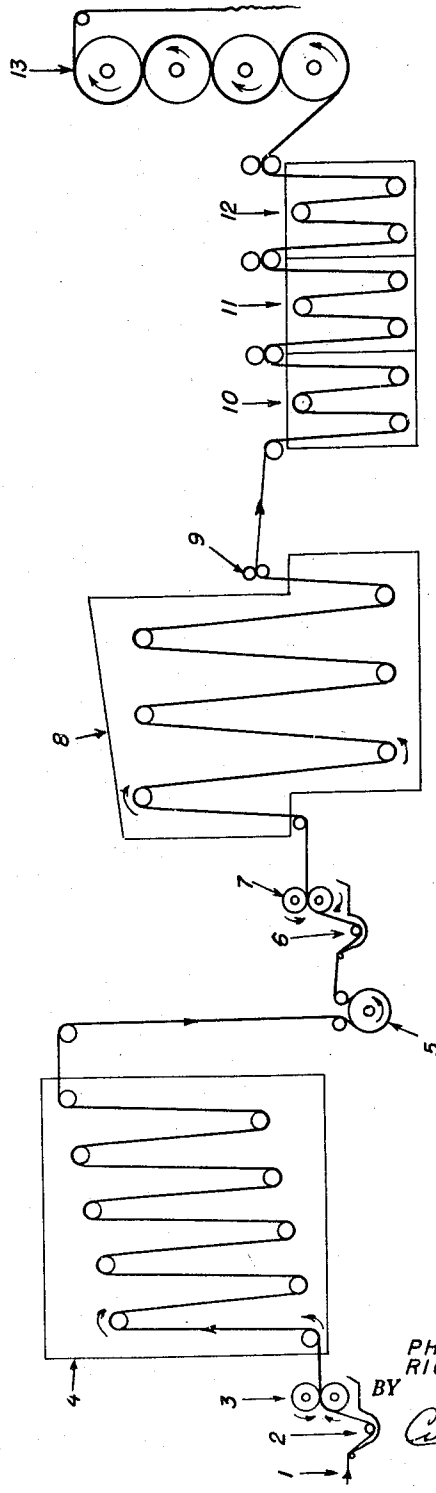
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PROCESS FOR DYEING TEXTILE FIBERS WITH VAT DYES

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PROCESS FOR DYEING 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 can 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, and the only one which has been adapted for the continuous dyeing 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 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 in some cases without running them 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 so that the cloth is dyed evenly throughout. 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 fiber 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 solution of the dye comes in contact with air, there is some surface dyeing which is objectionable.

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It is therefore an object of the present 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 for surface oxidation. A further object of the invention is to provide a continuous piece-goods dyeing process which will give results equally good or better than those obtained by the usual pad-jig method, particularly as to penetration and rub fastness, but which will obviate the handling of the goods and thereby speed up production and give more even dyeings.

We have found that textile material 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 which always involves the use of very dilute suspensions of the insoluble vat dye as more particularly illustrated in the following examples, and then passing the cloth 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 steam chamber, such as of the Mather-Platt type, whereby reduction and fixation of the color takes place quickly at temperatures of 212° F. and above. Because the dyeing is thus effected in one pass of the goods through the apparatus, and because the temperature at which the color is fixed is higher than heretofore employed, the time required to effect the dyeing is kept at a minimum. From the steam chamber, the goods is subjected to the usual oxidation, rinsing, soaping, rinsing and drying operations, all of which can be carried out in the continuous piece in the same manner as in the reduced-pad continuous dyeing processes.

In the accompanying drawing which forms a

part of this specification, a diagrammatic illustration of our dyeing process is given, showing the path of the textile to be dyed as it passes through the various steps in the process. In the drawing, the numeral 1 represents the textile which first passes through the padder 2 which contains the dispersed vat dye in pigment form, then through the nip rolls 3 which ordinarily constitute part of the padding apparatus; from there into the drier 4, then the goods is cooled in any conventional manner such as passing over the cooling can 5 so that the temperature of the goods as it passes through the caustic and hydrosulfite solution in the padder 6 is below that at which reduction normally takes place. After passing through the nip rolls 7 of the padder, the goods is immediately passed into an air-free steamer 8, then through the oxidizing, soaping and rinsing baths 10, 11 and 12, respectively, and finally passed over the drying rolls 13.

By passing the goods directly from the reducing solution into an atmosphere of steam from which all oxygen is excluded, no undesirable surface oxidation takes place and the reduction and fixation is effected so rapidly that there is no appreciable migration of the dye to the surface of the goods. By this process, the goods is dyed evenly throughout and maximum fastness of the dye is obtained.

After padding the goods with the unreduced vat color, it is preferably dried by passing it through a hot flue or over dry cans, for this permits better control in adding the chemical pad, that is, the caustic and reducing agent, and permits the amount of water remaining in the cloth to be held at a minimum. Because reduction is not brought about until the cloth enters the steam, and because the color is fixed at the higher temperatures, migration of the color in the goods is kept at a minimum.

If the chemical pad is applied to the wet pigmented goods, the moisture content of the final chemically 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 steaming has been found desirable in the production of level dyeings, for excessive moisture will tend to cause streaking, while insufficient water will result in poor development and therefore not a complete utilization of all of the color applied to the goods.

With most dyes, very satisfactory results are obtained when the moisture content of the goods as it enters the steam chamber is maintained between 50% and 100%, based on the weight of the fabric. With very difficulty vat-soluble colors, a higher moisture content may be desired. A higher moisture content may be employed on some goods than on others with equally good results, depending upon the texture and nature of the fibers being dyed.

The concentration of the caustic and 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 steam chamber is effected. Where the padder is set to retain 50% moisture from the chemical padding operation, a concentration of 4 ounces of caustic and 4 ounces of hydrosulfite per gallon is usually sufficient.

To prevent premature reduction of the dye on the fiber, the chemical padding operation, wherein the reducing agent and caustic are applied, is preferably carried out at temperatures not over

90° F., although temperatures up to 110° F. may be used.

The time of immersion in the chemical pad should be from 2 to 10 seconds, although a longer time may be required for certain types of fabrics. The time should be kept at the minimum required to obtain complete penetration of the chemical solution throughout the goods.

Following the chemical padding operation, it is desirable that the goods be passed as directly and quickly as possible into the steam chamber to avoid any tendency toward decomposition of the reducing agent, or oxidation of reduced dye. 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 steam chamber. This permits considerable leeway in leading the goods from the chemical pad to the steam chamber.

The steaming operation is preferably carried out at temperatures of from 212° to 240° F. At these temperatures substantially complete reduction and fixation are obtained in from 15 to 45 seconds. Longer periods of steaming may be employed when necessary. While periods of steaming longer than that actually required to effect complete reduction and fixation of the color may be employed without detriment to the resulting dye, it merely adds unnecessarily to the cost of the process, and is therefore to be avoided.

After development and fixation of the color in the steamer, the cloth is run into the usual oxidizing baths. Chromic acid, sodium perborate, hydrogen peroxide, or other oxidizing agent may be employed, or the material may be oxidized with air. After oxidation, the goods is soaped, rinsed and dried in the usual manner.

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

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

Example 1

Cloth is run through a padder set to retain 50% moisture containing the following pigment dyestuff suspension:

Ponsol Jade Green Paste (11.6% color solids) (C. I. 1101)	oz.	8
Water	gal.	1
"Alkanol" B (sodium alkyl-naphthalene sulfonate	oz.	0.15

while the padding bath is held at 140° F. This padded fabric is dried in a hot flue and padded with the following chemical pad, the squeeze rolls being set so that the goods will retain 50% moisture. The chemical pad consists of the following solution:

Caustic soda	oz.	4
Sodium hydrosulfite	oz.	4
Sodium chloride	oz.	4
Water	gal.	1

Temperature: 90° F. Time of immersion: 5 seconds.

The impregnated fabric is then passed through a substantially oxygen-free steam chamber (212°–215° F.) for a duration of 45 seconds. The dyestuff is in reduced form upon leaving the steamer, and is subsequently oxidized in the con-

ventional manner in 0.25% sodium bichromate solution containing 0.50% acetic acid. It is then rinsed in cold water, soaped, rinsed and dried.

Comparative dyeings were also made by developing the pigment vat color impregnated fabric on a jigger in the conventional manner.

Dyeings made by the new steam process were equal in penetration, shade and brilliancy, but showed superior fastness to laundering and scrubbing when compared with those products on the jigger in the conventional manner.

Example 2

A mixture of the following anthraquinone dyestuffs:

	Grams
The dyestuff of Example 1 of U. S. P. 2,212,029	3.34
The dyestuff of Example 1 of U. S. P. 995,936	0.12
The dye of Color Index No. 1151	0.97
Penta-anthrimide-carbazole	0.07

is made to 1 liter with water. This mixture is padded on herringbone twill fabric at 140° F., with the padder set for 50% moisture retention and the impregnated goods then dried.

The impregnated fabric is then padded with 50% moisture retention with a solution consisting of:

	Ounces per gallon
Caustic soda	1.5
Sodium hydrosulfite	4.0

At room temperature (70° F.).

The fabric is then passed into a substantially oxygen-free steam chamber for 1 minute, oxidized in the conventional manner in 0.25% chrome acetic acid solution, rinsed, soaped, rinsed and dried.

A comparative dyeing was made by developing the pigment impregnated fabric in a jigger containing 1.5 oz. per gallon caustic soda and 4 oz. per gallon sodium hydrosulfite at 140° F. for one-half hour, oxidizing and finishing as usual.

Olive-drab shades were obtained in both instances, and the dyeing obtained with steam process was fully equal in strength, shade and brilliancy versus the conventional jigger developed dyeing.

Example 3

A highly mercerized cotton fabric, such as is employed for the manufacture of men's very fine shirts, is dyed as follows:

Pad with 50% moisture retention the following vat dyestuff suspension:

Ponsol Blue GD paste (C. I. 1113) (color solids 16.4)	oz.	½
Water	gal.	1

Temperature: 140° F.

The pigmented goods is dried in a hot flue and padded with 50% moisture retention in a chemical solution containing:

	Ounces
Caustic soda	1.5
Sodium hydrosulfite	1.5
Sodium chloride	2

Temperature: 90° F. Time of immersion: 5 seconds.

The impregnated goods is then passed directly through an air-free steamer (212° F. to 215° F.) for a duration of 45 seconds. The dyestuff is in reduced form upon leaving the steamer, and is subsequently oxidized, rinsed, soaped, rinsed and dried.

Example 4

By this process Indian Head fabric, which is a highly mercerized and tightly woven fabric ordinarily very difficult to penetrate with dyes, can be dyed rapidly and very satisfactorily as follows:

The cloth is run through a padder set to retain 50% moisture, containing the following pigment dyestuff suspension which is held at 140° F.:

	Ounces per gallon
The dyestuff of Example 1 of U. S. P. 995,936	0.147
The dyestuff of Color Index #1101	0.116
The dyestuff of Example 5 of U. S. P. 1,705,023	0.0597
"Alkanol" B (sodium alkyl naphthalene sulfonate)	.0400

This padded fabric is dried in a hot flue and padded with the following chemical pad, the squeeze rolls being set so that the goods will retain 50% moisture.

Caustic soda	oz.	2
Sodium hydrosulfite	oz.	2
Sodium chloride	oz.	2
Water	gal.	1

Temperature: 90° F. Time of immersion: 5 seconds.

The impregnated fabric is then passed through a substantially oxygen-free steam chamber (212°–215° F.) for a duration of 45 seconds. The dyestuff is in reduced form upon leaving the steamer, and is subsequently oxidized in the conventional manner in 0.25% sodium bichromate solution containing 0.50% acetic acid. It is then rinsed in cool water, soaped, rinsed and dried.

The dyeings made by this process are of good strength, shade and brilliancy, but show excellent fastness to laundering and scrubbing and excellent penetration.

The dyeings developed by the new process are equal in strength, shade and brilliancy but superior in laundry and scrubbing fastness when compared with dyeings developed by the conventional pad-jig process.

Any of the cellulose or other fibers normally dyed with vat dyes may be dyed by the processes above described. Viscose rayon, or cotton and viscose rayon mixtures, are preferably dyed by the process of Example 1 or Example 3.

The process of this invention is applicable for the dyeing of warps, skeins or any other form of textile fiber which can be introduced into the pigment pad, chemical pad and development chamber in a continuous manner. It is of course understood that other vat dyes than those specifically employed in the above examples may be dyed by this method, such as the thioindigos, sulfur colors, and any others which may be dyed by the vat dye method.

In place of the sodium hydrosulfite, other reducing agents which operate in a similar manner may be employed. With milder reducing agents, a longer time of development in the steam chamber is required, as is also the case with certain more difficultly reducible vat colors.

The time required for complete reduction and development in the steam chamber will depend upon the ease with which the color is reduced and upon the type of fiber being dyed.

The cloth, on entering the development chamber, is preferably heated quickly to the tempera-

ture of the chamber, and the heating should be carried out in such a manner that it is uniform on both sides of the fabric, and the steam should flow through the chamber in such a manner as to remove the decomposition products of the reducing agents at a satisfactory rate. Formation or deposition of moisture, as condensed on the fabric, should be avoided.

The process as above described provides a very satisfactory method for dyeing goods in a continuous piece and for obtaining dyeings of good penetration and which have maximum fastness properties, particularly with regard to abrasion and laundry tests. The process permits a material saving in the volume of dyeing liquid required, and, since no developing solution is employed, there is no loss of dye in the finally discarded developing bath, for substantially all of the dye which is applied to the fiber is fixed therein. The process permits the dyeing of textile fibers in very even pastel shades, and also permits the application of even the most difficultly reducible vat dyes in heavy shades.

We claim:

1. A rapid continuous process for dyeing textile fibers with vat dyes which comprises padding the fiber with a dilute aqueous suspension of an unreduced vat dye which does not contain a thickening agent, padding the fiber containing dye with an alkaline solution of sodium hydrosulfite for a period of from 2 to 10 seconds and at a temperature below that at which any material reduction of the dye takes place, the concentration of the alkaline solution of the sodium hydrosulfite being such that at normal vatting temperatures it will effect reduction of all of the dye in the fiber, and immediately subjecting the fiber so padded to the action of steam in the absence of oxygen for a period of from 15 to 45 seconds to effect reduction and fixation of the dye in the fiber.

2. A rapid continuous process for dyeing textile fibers with vat dyes, which comprises padding the fiber with a dilute aqueous suspension of an unreduced vat dye which does not contain a thickening agent, drying the fiber so padded, padding the fabric containing the dye for a period of from 2 to 10 seconds with from 50% to 100%, based on the weight of the fabric, of a caustic alkali solution of sodium hydrosulfite containing from 1.5 to 4 ounces of each of the caustic and hydrosulfite per gallon of solution maintained at temperatures not over 90° F., and, as fast as padded, immediately subjecting the fiber padded with the color, caustic alkali and hydrosulfite to the action of steam in the absence of oxygen for a period of from 15 to 45 seconds to effect reduction and fixation of the dye in the fiber.

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