

## UNITED STATES PATENT OFFICE

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## PROCESS OF DYEING

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This invention relates to vat dyeing processes.

It is an object of this invention to provide an improved process for dyeing with vat dyestuffs, which is particularly adapted for continuous dye-  
5 ing.

It is a further object of this invention to provide an improved process for dyeing with mixtures of vat dyestuffs.

Other and further objects of this invention will  
10 appear as the description proceeds.

In dyeing with vat dyestuffs, trouble is often experienced due to the fast rate of absorption of the dyestuff on the fiber. Since not all parts of a given piece of cloth, or package of yarn,  
15 are exposed to the vat at the same moment, the first portions to contact the vat get the benefit of a strong vat while some others come in contact with a partially exhausted vat. The result is a spotty and unevenly dyed piece of fabric or package of yarn.  
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In dyeing with a vat dye on a continuous machine, wherein the fabric is run off a roll, dipped continuously into a vat and then wound upon another roll or passed through another bath  
25 (the re-reducing bath) similar difficulties are experienced. As the fabric passes through the vat, the dye is taken up and the vat is gradually being exhausted. To overcome this, further additions of dyestuff to the bath are necessary. It is clear, however, that unless the subsequent  
30 additions of dyestuff are carefully regulated and timed to the rate of exhaustion of the vat, certain parts of the bolt or roll will be dyed stronger than others.

Then too, in order to obtain the desired shade it is often necessary to use a mixture of dyes whose rates of dyeing are quite different. Since the fabric remains in the dyebox only a very short time, much less than is required for complete exhaustion, it will be apparent that the  
40 more rapid-dyeing color will in this case be exhausted more rapidly and the shade of the dye cloth will change as the roll proceeds through the vat. In order to overcome this, it is necessary to make additions of the more rapid dye-  
45 ing component and constant adjustment is necessary. Such adjustment becomes more difficult the more difference there is between the rates of dyeing of the components.

It has accordingly been the custom in the art to add a retardant to the vat. The retardant is an agent which diminishes the rate of absorption of the dye on the fabric. When dyeing with a plurality of vat dyestuffs, the retardant will  
55 generally slow down the rate of the faster dye

relatively more than the rate of slower dye, and will thereby diminish the difference in the dyeing rates of the individual components of the mixture and facilitate adjustment of the mixture as the fabric proceeds through the vat. In dye-  
5 ing with a single color in the continuous process, the retardant likewise facilitates control of the strength of the vat, thereby rendering the dyeings more uniform.

In package dyeing, the retardant is also bene-  
10 ficial in that it delays exhaustion until practically all portions of the package have become wetted by the coloring solution. The result is improved penetration and more level dyeings.

As such retardants in the art, soap and glue  
15 have been employed. The operation of these, however, is not always satisfactory. In the first place the degree of retardation is relatively small; in the second place there are many dyestuffs as to which the mentioned retardants fail alto-  
20 gether.

We have now found that cellulose ethers which are soluble or dispersible in the vat are excellently adapted for the purpose mentioned. Their action is generally considerably more  
25 marked than that of glue or soap, and their range of applicability is much wider.

Any cellulose ether which forms a stable colloidal solution in dilute caustic at dyeing temperatures (from room temperature up to about  
30 140 or 160° F.) may be used for our purpose. Among these may be mentioned the various ethers of cellulose which contain in the non-cellulosic radical a group tending to make the molecule soluble in alkali; for instance, cellulose eth-  
35 ers containing in the non-cellulosic radical sulfonic, carboxy or hydroxy groups.

Compounds of this type are described in U. S. Patents 1,722,927 and 1,682,382; see also, "Technology of cellulose ethers" by E. C. Worden, vol. 40  
3, (Chem. Cat. Co., New York, 1933). The stage at which the retardant is added is unimportant so long as it is present in the vat or on the fibre during dyeing.

As the simplest and commonest example of  
45 a cellulose ether which is soluble in alkaline solution may be mentioned sodium cellulose glycollate. This one is generally prepared from sodium cellulose and the sodium salt of chloroacetic acid, and is a relatively inexpensive commercial ar-  
50 ticle. Other ethers, however, of cellulose may be employed successfully, provided they are soluble in the vat without decomposition, for instance, the ether of cellulose and chloropropionic acid or its alkali-metal or ammonium salts.  
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Without limiting our invention to any particular procedure, the following examples are given to illustrate our preferred mode of operation. Parts are by weight.

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### Example I

2.5 parts of trichloro-indanthrone double paste and 2.5 parts of flavanthrone double paste are mixed with 31.5 parts of 31° Bé. caustic soda; 10 214 parts of water are added and the temperature is adjusted to 120° F. 7.5 parts of sodium hydrosulfite are now stirred in. After 15 minutes 160 parts of a 2.5% aqueous solution (or sol) of sodium cellulose glycollate are added and then 15 1590 parts of water, keeping the temperature of the vat at 120° F. The vat is now ready for dyeing 100 parts of yarn or fabric by any desired process and will be found to give the improved results above discussed. Similar im- 20 proved results are observable when one uses a mixture of trichloro-indanthrone and the dyestuff described in Example 3 of U. S. P. 1,329,435.

### Example II

25 2.5 parts of a 20% paste of the dyestuff described in Ex. 3 of U. S. P. 1,329,435 are mixed with 31.5 parts of 31° Bé. caustic soda and 217 parts water. The temperature is adjusted to 140° F. and 7.5 parts of sodium hydrosulfite are 30 stirred in. After 15 minutes 100 parts of 2.5% aqueous sodium cellulose glycollate are added and then 1750 parts of water, the temperature being maintained at 140° F. The vat is now ready for dyeing 100 parts (more or less according to the 35 depth of shade desired) of yarn or fabric by any standard process.

Instead of the sodium salt of cellulose-glycollic acid, any other alkali-soluble salt may be employed, for instance, the potassium or ammonium 40 salt. The free acid form of this compound may also be used. The quantity of retardant employed may vary within wide limits. Very marked retardation has been obtained with quantities varying from 1 to 10% by weight based on 45 the weight of the fibre.

It will be understood that the dyes mentioned are only illustrative, since our novel retardant gives excellent results with a great many other vat dyestuffs when used individually or in com- 50 bination.

If desired, the retardant may be sprayed or impregnated upon the fiber instead of adding it to the vat. The latter then may be prepared of the usual ingredients in the ordinary manner.

55 Many other variations and modifications are possible in our preferred embodiment above set forth without departing from the spirit of this invention.

As already mentioned above, our invention may 60 be practiced with a cellulose ether which is actually soluble in alkaline medium, or with one which merely gives a stable colloidal solution. Therefore, in the claims below we shall use the expression "alkali-dispersible" as a generic term to

cover both the property of complete or theoretical solubility and the property of yielding a stable colloidal solution.

We claim:

1. A process of dyeing fiber with a vat dyestuff 5 which comprises applying the dyestuff solution to the fiber in the presence of a cellulose ether which is adapted to be dispersed in alkaline solution.

2. A process of dyeing fiber with a vat dyestuff which comprises applying the dyestuff solution to 10 the fiber in the presence of a cellulose ether which is adapted to form a colloidal suspension or solution in dilute aqueous alkali.

3. A process of dyeing fiber with a vat dyestuff which comprises applying the dyestuff solution 15 to the fiber in the presence of a cellulose ether which is a protective colloid and is soluble in alkali.

4. A process of dyeing fiber with a vat dyestuff which comprises applying the dyestuff solution 20 to the fiber in the presence of a cellulose ether which carries in the non-cellulosic radical a group tending to make the molecule soluble in alkali.

5. A process of dyeing fiber with a vat dye- 25 stuff which comprises applying the dyestuff solution to the fiber in the presence of a cellulose ether which contains a solubilizing radical selected from the group consisting of sulfo, carboxy and hydroxy. 30

6. A process of dyeing fiber with a vat dyestuff which comprises applying the dyestuff solution to the fiber in the presence of cellulose-glycollic compound selected from the group consisting of 35 the free acid and an alkali-soluble salt thereof. 35

7. A process of dyeing fiber with a vat dyestuff which comprises applying the dyestuff solution to the fiber in the presence of sodium cellulose glycollate.

8. In the process of dyeing fabric with a vat 40 dyestuff by the continuous process, the improvement which comprises incorporating into the dye bath an alkali-dispersible ether of cellulose.

9. In the process of dyeing fabric with a vat 45 dyestuff by the continuous process, the improvement which comprises incorporating into the dye bath the cellulose ether of glycollic acid or a water soluble salt thereof.

10. In the process of dyeing fabric with a plurality of vat dyestuffs the improvement which 50 comprises effecting the dyeing in the presence of an alkali-dispersible ether of cellulose.

11. In the process of dyeing fabric with a plurality of vat dyestuffs the improvement which comprises effecting the dyeing in the presence of 55 a salt of cellulose glycollic acid.

12. A retardant for vat dyestuffs which comprises a cellulose-glycollic compound selected from the group consisting of the free acid and alkali-soluble salts thereof as principal ingre- 60 dient.

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