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DYEING OF POLYESTERS WITH VAT DYES

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This invention relates to a method of dyeing polyester fibers with vat dyes.

Polyesters such as polyglycol terephthalate have obtained extensive commercial use but have presented a considerable problem in dyeing. Only certain dyes have been useful. Unfortunately, vat dyes with their great fastness to light, washing and other treatments, have failed to dye polyester fibers, and so this important field of dyestuffs has been unusable. According to the present invention, we have found that it is possible to dye polyester fibers with vat dyestuffs under certain very definite conditions. The most important is the pH of the dye bath which must be between 6 and 8, and preferably between 6.5 and 7.5. At this pH, vat dyestuffs are reduced to their leuco compounds in the form of so-called vat acids in which the hydroxyl groups are free rather than the sodium salts which are produced in the ordinary vats. It is not known why the vat acids so-called—for they are not true acids except in the sense that phenols are sometimes called acids because they react with strong bases to form salts—behave in such a satisfactory manner in dyeing polyester fibers where they will not dye practically cellulose as cotton and viscose rayon. It is not known what the reason is for this anomalous behavior.

The present invention requires the definite pH range. However, it is not important to the operativeness of the process how the range is obtained and maintained. It is perfectly possible to use strong alkali aqueous solutions of caustic soda. However, this requires rather careful supervision of the dyeing process because the pH of the dye bath tends to change as the reducing agent, normally sodium hydrosulfite, becomes oxidized to sodium bisulfate and thus reduces the pH. Therefore, in the preferred embodiment of the present invention buffers are used to automatically maintain the desired pH range; preferably these are polyphosphate buffers such as sodium tripolyphosphate or sodium tetraphosphate. When a buffer is used, it is not necessary to provide careful supervision and control in order to maintain the pH. Of the two buffers, sodium tripolyphosphate is somewhat the better.

The second condition which is necessary to the present invention is a high temperature. Satisfactory results are not obtained if the dyeing is effected below the boiling point of water. Even at the boil the dyeing is slow and with some dyes the levelness leaves something to be desired. Excellent results are obtainable at higher temperatures, preferably from about 250° F. to 290° F. The upper limit on temperatures is not critical and is determined by a temperature which the polyester fibers will stand. As a practical matter, temperatures above 325° F. are too high and tend to damage the fibers.

The invention will be described more fully in conjunction with the following specific examples, the parts being by weight unless otherwise specified.

Example 1

A staple polyglycol-terephthalate yarn normally sold

under the trade-name "Dacron" was dyed using 10% by weight of 6,6'-dichloro-4,4'-dimethyl-2,2'-bis thionaphthene indigo in a bath having a 10:1 water ratio and containing 6% sodium tripolyphosphate and 4% sodium hydrosulfite. The dye was then oxidized by boiling with a dilute 13% hydrogen peroxide solution containing 1% acetic acid. The dyeing took only a few minutes at 290° F. and a bright pink shade resulted. Microscopic examination showed that the fibers were well penetrated. After dyeing the staple was washed with water and then with a solution of sodium dodecyl sulfate in accordance with standard conventional practice.

Example 2

The procedure of Example 1 was followed at 250° F. A good dyeing was obtainable but the dyeing time was about 12 times as long.

Example 3

The procedure of Example 1 was followed with "Dacron" filament yarn using 3% sodium tripolyphosphate and 2% sodium hydrosulfite. The same results were obtained as with the staple yarn, microscopic examination showing good penetration.

Example 4

The procedure of Example 3 was followed but the dyeing was effected at a lower temperature of 250° F. The results were the same but the dyeing time was very markedly extended.

Example 5

The procedure of Example 1 was followed but instead of using the tripolyphosphate, sufficient amount of 30° Bé. aqueous sodium hydroxide was employed to bring the bath to a pH of between 6.5 and 7.5, the results being identical.

Example 6

The procedure of Example 5 was followed using filament yarn instead of staple and employing 2% sodium hydrosulfite instead of 4%. A smaller amount of the dilute caustic soda solution was of course required to maintain the pH. The results were the same as in Example 3.

Example 7

The procedure of Example 1 was followed but the pink vat dyestuff was replaced by a corresponding amount of Vat Green Color Index No. 1184. A well-penetrated green dyeing resulted.

Example 8

The procedure of Example 7 was repeated at 250° F. The results obtained were the same but the dyeing time was greatly extended.

Example 9

5 parts of a skein of Dacron staple yarn was dyed in a bath containing 10% of Jade Green (dimethoxydibenzanthrone), 3% sodium tripolyphosphate and 2% sodium hydrosulfite, the bath fiber ratio being 10:1. The dyeing was complete in a few minutes at 290° F. and then the yarn was removed and boiled in a 1% aqueous sodium carbonate solution and oxidized by boiling with dilute, (0.3%) hydrogen peroxide solution containing 1% acetic acid. A deep blue green color resulted which was fast to washing when boiled for 10 minutes in a bath containing 0.25% sodium dodecyl sulfate and 0.25% acetic acid.

Example 10

The procedure of Example 1 was followed except that the temperature was reduced to 250° F. and the dyeing time increased 12 times. A blue green dyeing was obtained which on microscopical examination showed that

the dye had penetrated into the fibers. The dyeing, however, was not as strong and the penetration not as deep as in Example 1.

Example 11

5 parts of a skein of Dacron staple yarn was dyed in a bath containing 5% Hydron Pink FF (6,6'-dichloro-4,4'-dimethyl-2,2'-bis thionaphthene indigo), 3% sodium tripolyphosphate, 2% sodium hydrosulfite and 1% aluminum chloride with 6 mols of water of crystallization, the bath having a bath-fiber ratio of 10:1. The dyeing proceeded very rapidly at 290° F. After completion of the dyeing the skein was removed from the bath and boiled with 1% aqueous sodium carbonate solution followed by oxidizing by boiling with 1 part of a 3% hydrogen peroxide solution containing 1% acetic acid. The skein was then washed in a boiling water solution containing 0.25% sodium dodecyl sulfate and 0.25% acetic acid. A bright pink dyeing resulted, the fibers being well penetrated when examined under the microscope.

Example 12

The procedure of Example 11 was followed, substituting for the Hydron Pink the blue dye CI No. 1184 and replacing the aluminum chloride with an equivalent amount of zirconium oxychloride. A deep blue dyeing resulted, showing good penetration.

We claim:

1. A process of dyeing fibrous polyesters of a glycol and a polycarboxylic acid with vat dyes, which comprises heating the said polyester fibers in an aqueous dyebath containing an unesterified leuco vat dye, a reducing agent and sodium tripolyphosphate, and having a pH between 6.5 and 7.5, at a temperature between the boiling point of water and 290° F., the pH of the aqueous dyebath during dyeing being maintained between 6.5 and 7.5 by means of the sodium tripolyphosphate buffer, and oxidizing the dyestuff on the fiber after dyeing is complete.

2. The process of claim 1 wherein said polyester fiber is a poly glycol-terephthalate fiber.

UNITED STATES PATENTS

References Cited in the file of this patent

1,900,168	Dreyfus	Mar. 7, 1933
1,900,172	Ellis	Mar. 7, 1933
2,627,449	Luttringhaus	Feb. 3, 1953

OTHER REFERENCES

"Chem. Tech. of Dyeing and Printing," by L. Diserens, published New York City, 1948, by Reinhold Publishing Corporation, pages 135 and 136.

American Dyestuff Reporter for January 22, 1951, pages P51 to P54 inclusive.

American Dyestuff Reporter for October 15, 1951, pages P681 to P684 inclusive.