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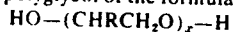
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[54] **PROCESS FOR THE PRODUCTION OF LEVEL
 END-UNIFORM DYEINGS WITH WATER-
 SOLUBLE SULPHURIC ACID ESTERS OF
 LEUCO-VAT DYESTUFFS**
4 Claims, No Drawings

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35, 21, 93

ABSTRACT: A process for the production of consistently level and end-uniform dyeings on textile materials selected from the group consisting of cotton and of mixtures of cotton and polyesters by padding with sulfuric acid esters of leuco-vat dyestuffs, wherein the padding is carried out in the presence of a polyglycol of the formula



in which R is a member selected from the group consisting of hydrogen and methyl and x is an integer, and which has an average molecular weight within the range of 1,000 to 20,000.

PROCESS FOR THE PRODUCTION OF LEVEL END-UNIFORM DYEINGS WITH WATER-SOLUBLE SULPHURIC ACID ESTERS OF LEUCO-VAT DYE STUFFS

It is known that water-soluble sulfuric acid esters of different leuco-vat dyestuffs have different affinities for vegetable fibers. The different affinities have an unfavorable effect in padding fabrics of vegetable fibers in that dyeings are obtained of which the depth of color is dissimilar at the two ends of the fabric. This difference is especially troublesome in padding with mixtures of weakly and strongly substantive sulfuric acid esters of leuco-vat dyestuffs. Owing to the fact that the more substantive dyestuff is more rapidly absorbed changes in shade occur during the dyeing operation, which, especially in dyeing great lengths, lead to great differences in shade between the beginning and the end of the dyed material.

As is known, attempts have been made to solve this problem by using a smaller padding frame and by increasing the concentrations of the individual dyestuffs in the liquor subsequently added in accordance with their substantivities. However, these expedients have in practice been found to overcome the difficulties only partially.

It is also known to attack this problem by the addition of compounds affecting dyestuff affinity to the padding bath. By means of these known substances, that are also called levelling agents, the substantivity towards vegetable fibers is reduced or adjusted. In this manner it is indeed possible to produce dyeings that are improved with respect to uniformity at the ends of the fabric. As suitable substances for this purpose there have been proposed, for example, proteins, longchain quaternary ammonium compounds, polyvinyl-pyrrolidones and ethylene oxide addition, products based on aliphatic alcohols, alkyl-phenols, fatty acids and fatty amines and having a content of about 5 to 30 ethylene oxide residues.

However, the hitherto recommended compounds affecting dyestuff affinity have not been satisfactory in all respects. Either their action has not been sufficient to ensure unobjectionable end-uniform dyeings in padding large pieces or undesired side effects to occur when the proposed substances are used. A large proportion of the levelling agents in practice cause an especially troublesome foam formation. Moreover, with certain sulfuric acid esters of leuco-vat dyestuffs they may form association compounds that are sparingly soluble in water. Such precipitates impair the dyeing yield and uniformity, and this disadvantage cannot be completely overcome by the use of dispersing agents. Thus, for example, most ethylene oxide addition products can only be removed with difficulty from the fabric after dyeing, and, owing to their surface-active properties, they injuriously affect subsequent textile treatment processes, for example, hydrophobization.

The present invention is based on the observation that satisfactory, consistently level and end-uniform dyeings are obtained by using a padding bath containing a polyglycol of the formula



in which R is a hydrogen atom or a methyl group, and x is a whole number, and which has a molecular weight within the range of 1,000 to 10,000.

As polyglycols there are used polyethylene glycols or polypropylene glycols. There are also suitable mixtures of polyethylene and polypropylene glycols, and copolymers of ethylene oxide and propylene oxide. In practice there are preferably used ordinary commercial mixtures of polyglycols having different molecular weights. Especially good results are obtained by using polyethylene glycols having average molecular weights within the range of from 1,000 to 10,000.

The padding bath preferably contains, per liter, 0.5 to 10 grams of a polyglycol or polyglycols used in the invention. However, smaller or larger quantities may be used. In addition to the polyglycols and the sulfuric acid esters of leuco-vat dyestuffs the padding baths may contain the usual assistants. However, the addition of dispersing agents is not necessary. The padding may be carried out at various temperatures, and is preferably carried out at room temperature. Fluctuations in temperature do not adversely affect the end uniformity.

The process of the invention is suitable for dyeing fabrics of vegetable fibers and also for dyeing mixed fabrics containing vegetable fibers.

The following examples illustrate the invention, the parts being by weight:

EXAMPLE 1

In a bath containing, per 1,000 parts, 2 parts of the highly substantive dyestuff of Color Index (2nd Edition) No. 59,826, 5 parts of sodium nitrate and 2 parts of polyethylene glycol having an average molecular weight of 4,000, a cotton fabric is padded at room temperature with a squeezing effect of about 80 percent. The material is then dried, developed continuously in a bath containing 3.4 percent of sulfuric acid, and finished in the usual manner. Even with very great lengths no difference between the depths of the dyeings at the beginning and end portions of the fabric could be detected.

Instead of polyethylene glycol having a molecular weight of 4,000, there may be used polyethylene glycols having molecular weights within the range of from 1,000 to 20,000 or a polypropylene glycol having a molecular weight of 1,200.

There can be used with equal success in this example the dyestuffs of Color Index Nos. 59,051, 60,011, 69,526 and 70,801.

By using in this example, instead of 2 parts of the dyestuff of Color Index No. 59,826, 10 parts of the dyestuff of Color Index No. 59,051 there are likewise obtained level and end-uniform dyeings. If the polyglycol is replaced by the same quantity of a condensation product of 1 mol of nonyl-phenol and 10 mols of ethylene oxide precipitation takes place in the padding bath, which causes an unlevel and spotty dyeing.

If in this example the polyethylene glycol used is replaced by the same quantity of a polyethylene glycol having an average molecular weight of 200, there is obtained an effect that is considerably less favorable with respect to end-uniformity.

EXAMPLE 2

A solution containing, per 1,000 parts, 5 parts of the dyestuff of Color Index No. 69,526, 0.3 part of the sulfuric acid ester of the leuco-vat dyestuff of French specification No. 1,037,419, 0.1 part of the dyestuff of Color Index No. 59,706, 50 parts of urea, 30 parts of an aqueous solution of 5 percent of the sodium salt of polymeric methacrylic acid, 2 parts of a polyethylene glycol having an average molecular weight of 4,000, 0.1 part of sodium metavanadate, 8 parts of ammonium thiocyanate and 3 parts of sodium chlorate, is padded at room temperature onto 5,000 meters of a mixed fabric of polyester and cotton (67/33) with a squeezing effect of about 60 percent. After being dried, the mixed fabric is subjected for 1 minute to a dry heat treatment at 200° C., and then finished in the usual manner. The whole dyeing process is carried out in a fully continuous manner. There is obtained a level grey dyeing that, in spite of the differing substantivities of the dyestuffs, exhibits no change in the depth or shade of the dyeing.

The padding bath used in this example has a considerably lower foaming capacity than a padding bath that contains 1 part of a condensation product of 1 mol of nonyl-phenol and 10 mols of ethylene oxide.

EXAMPLE 3

In a padding bath containing, per 1,000 parts, 1 part of the highly substantive dyestuff of Color Index No. 59,826, 1 part of the dyestuff of Color Index No. 69,826 having a lower substantivity, 8 parts of sodium nitrate and 4 parts of a polyethylene glycol having an average molecular weight of 2,000, a cotton fabric is padded at 40° C. with a squeezing effect of about 80 percent. The dyeing is developed in a continuous manner as described in example 1. The turquoise dyeing exhibits no change in color from the beginning to the end.

EXAMPLE 4

7.5 parts of the dyestuff of Color Index No. 67,001, 3.5 part of the dyestuff obtainable according to example 8 of French Specification No. 1,167,704, 1.5 parts of the sodium salt of polymeric methacrylic acid and 2 parts of a polyethylene glycol having an average molecular weight of 4,000 are dissolved in 1,000 parts of water and padded at room temperature onto a mixed fabric of polyester and cotton (67/33) with a squeezing effect of about 60 percent. After being dried, the mixed fabric is subjected for 1 minute to a dry heat treatment at 200° C. Then the mixed fabric is passes for 3 seconds at room temperature through a bath containing 40 parts of 96 percent sulfuric acid and 1 part of sodium nitrate in 1,000 parts of water. Finally the dyeing is finished in the usual manner. The whole dyeing process is carried out in a fully continuous manner. There is obtained a fully level dyeing. Even when dyeing several thousands of meters of mixed fabric, no change in the depth or shade of the dyeing can be noticed. By using higher or different padding temperatures the levelness of the dyeing is not impaired.

If the dyeing is developed, after the dry-heat treatment, for 10 seconds at 60-70 ° C. is a dyebath containing 3 parts of sodium chlorate, 10 parts of 96 percent of sulfuric acid and 0.5 part of sodium metavanadate dissolved in 1,000 parts of water, excellent shades are also obtained.

Instead of the polyethylene glycol used in the above exam-

ple 4 it is also possible to use 5 parts of a polyethylene glycol having an average molecular weight of 1,000. In this case the same results are obtained.

We claim:

1. A process for the production of consistently level and end-uniform dyeings on textile materials selected from the group consisting of cotton and of mixtures of cotton and polyesters by padding with sulfuric acid esters of leuco-vat dyestuffs, wherein the padding is carried out is the presence of a polyglycol of the formula



in which R is a member selected from the group consisting of hydrogen and methyl and x is an integer, and which has an average molecular weight within the range of 1,000 to 20,000.

2. A process as defined in claim 1, wherein the polyglycol has an average molecular weight within the range of 1,000 to 10,000.

3. A process as defined in claim 1, wherein R is hydrogen and the polyglycol has an average molecular weight within the range of 1,000 to 10,000.

4. A process as claimed in claim 1, wherein R is hydrogen and the polyglycol has an average molecular weight within the range of 1,000 to 10,000 and wherein the polyglycol is present in concentration within the range of 0.5 to 10 grams per liter.

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