

[54] DYEING SYNTHETIC AND NATURAL FIBERS WITH THE LIQUID AMMONIA AND CHLOROFORM OR METHYLENE CHLORIDE SOLUTION OF A DYE

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[58] Field of Search..... 8/82, 162, 174

[56]

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[57]

ABSTRACT

Process for dyeing textile materials of natural or synthetic fibers alone or in mixture with each other, by treating the fibrous material with a solution of suitable dyestuffs in anhydrous, liquid ammonia alone or in mixture with low boiling organic solvents, within the range below the boiling point of the dyebath, and fixing the dyestuffs applied after removal of the ammonia remaining in the material in known manner typical for the dyestuffs or the dyegoods used.

12 Claims, No Drawings

DYEING SYNTHETIC AND NATURAL FIBERS WITH THE LIQUID AMMONIA AND CHLOROFORM OR METHYLENE CHLORIDE SOLUTION OF A DYE

Liquid ammonia has been used for several years in the textile finishing industry for treating cellulose fibers: Thus, for example, sewing yarns of cotton are treated according to the so-called Prograde-Process (J. & P. Coats Ltd., Paisley) with liquid ammonia during $\frac{1}{2}$ - $\frac{3}{4}$ seconds at a temperature below -33°C . The yarn is then passed through a hot water trough where it is drawn at about 93°C . The material is then dried by means of hot air and wound up. The yarn is thereby given a higher strength, a better stability in length, an improved absorbing power for dyestuff and a higher resistance towards thermal strain.

Furthermore, there has been developed the so-called Tedeco-Process (Norwegian Textile Research Institute in cooperation with the Norwegian Central Institute for Industrial Research) according to which piece goods of cotton are subjected for about 10 seconds to the influence of liquid ammonia. The ammonia absorbed is then removed by passing the goods over heated cylinders and recovered for further utilization. Due to this treatment the goods shrink, show an improved dyeability and appearance, a higher resistance to wear and tear and a higher stability of dimensions. When in addition to this treatment the textile goods are drawn, a higher strength is obtained. Mixtures of polyester and cotton fibers can be treated likewise. The properties of the cotton component are thereby improved without impairment of those of the polyester fiber.

In the field of textile dyeing there is a rather considerable tendency to abstain from using water hitherto nearly exclusively used as dyeing medium in dyeing and padding baths and to use instead of it other solvents, mainly organic solvents. Based on the experience made in the dry cleaning, for example chlorinated hydrocarbons are proposed. These processes have quite a number of great advantages.

There are no costs for procuring and preparing service water and for the purification of waste-water which becomes more and more difficult, the costs for the energy are lower and the dyeing time is substantially reduced which is of increasing importance considering the continuously rising wages. In contradistinction thereto, the costs for recovering the solvents do not matter. These advantages are not restricted to organic solvents, inorganic solvents may principally be taken into consideration, too.

The present invention is based on the observation that liquid ammonia is a good solvent for dyestuffs. Thus, for example, disperse, pigment, reactive, acid, metal complex and cationic dyestuffs irrespective of their chemical constitution can be dissolved in liquid ammonia in concentrations which are generally sufficient for dyeing operations. Such dyestuff solutions for example are suitable as treating baths for dipping, padding and spraying of textile material in the form of slubbing, cables, filaments, knitted, woven and non-woven fabrics.

The present invention concerns a process for dyeing textile material made from natural or synthetic fibers alone or in mixture with each other wherein the fiber material is treated with a solution of suitable dyestuffs in anhydrous, liquid ammonia alone or in mixture with

low boiling organic solvents within the range below the boiling point of the dyeing bath, and the dyestuffs are fixed after removal of the ammonia remaining in the goods in known manner which is typical for the dyestuffs or dyegoods used.

The plants in which the treatment of the textile materials is to be carried out are adapted to the physical and chemical properties of the liquid ammonia. Thus, for example, the materials used have to resist the low temperatures below the boiling point of the ammonia of -33°C and also a possible dissolving effect or corrosion by the ammonia. The entire dyeing plant consists mainly of a dissolving and preparing vessel with stirring arrangement and filter for preparing the dye bath, a liquor application machine, for example a padding foulard with trough or a spraying device, a heatable mechanism such as a hollow roller to remove the ammonia after the treatment, and a recovering device for the ammonia. The afore-mentioned dyeing apparatus is made air-tight by inlet and outlet roller locks.

The experiences of the compressor cooling technique and the dry cleaning, and the latest knowledge of the dyeing technique with solvents as well as of the aforementioned pretreatment of the cotton with liquid ammonia have been utilized in the present invention.

The dyestuff selected according to the fibers to be dyed and eventually according to its solubility is dissolved while stirring with a stirring mechanism in the preparing vessel outlined hereinbefore. When this dyestuff is in the form of a commercial product it generally still contains extenders for setting a constant portion of the coloring substance which agents are often insoluble in liquid anhydrous ammonia. When the dissolving process is finished, the solution of the dyestuff is passed through a filter device to the storage vessel, for example the pad trough, the extender remaining in the filter. When the dyestuff does not contain an extender for example in the case of pigments or dyestuffs without any auxiliary agents, a filtration is unnecessary.

With the dyestuff solution thus prepared the textile material is impregnated either by dipping and squeezing, dipping and sucking off or spraying. It may be that the liquid ammonia contains portions of extremely low boiling organic solvents which thereby form azeotropic mixtures and may be jointly regenerated as it is known from the refrigeration technique.

After the impregnation of the dyegoods, the ammonia is removed by allowing it to evaporate for example by passing it over a heated roller or simply by reducing the pressure in the closed dyeing vessel, and fed back in the recovering system, eventually by compression and cooling, in liquid form into the circulation.

The textile material leaves the dyeing plant in dry condition and is further treated for its mostly thermal fixing depending on the type of the fiber and the dyestuff used.

If according to the claimed process reactive dyestuffs are applied on cellulose fibers, the application and fixation may be effected by one of the known techniques, for example the pad-batch, two bath-pad-steaming or wet-fixation process.

Disperse dyestuffs on polyester fibers are fixed by pressure steam, solvent steam or thermosoling.

On polyamide fibers steaming with saturated steam also suffices.

Cationic (basic) dyestuffs on acrylic or acid-modified polyester fibers are fixed by steaming.

In the case of fiber mixtures, for example fibers made from polyester and cellulose, the disperse dyestuff on the polyester fiber portion is fixed thermosoling, the reactive dyestuff on the cellulose fiber portion is fixed by one of the known two-phase techniques.

Unfixed dyestuff portions have to be removed after fixing if necessary by an after-treatment.

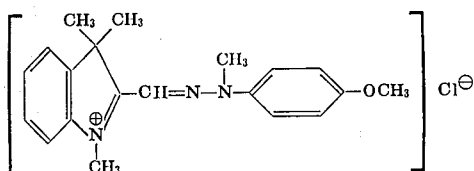
The following textile materials can be dyed according to the process of the present invention: natural and regenerated cellulose fibers, polyester and modified polyester fibers, polyamide and modified polyamide, polyurethane, polyolefin and modified polyolefin fibers as well as polyacrylic fibers, and other fibers which can be dyed with disperse, reactive and cationic dyestuffs.

In the case of the application of mixtures from liquid ammonia and organic solvents as dyeing medium there are especially used as solvent-mixture component according to the present invention alcohols such as methanol, ethanol, iso-propanol and n-butanol, furthermore acetone, dimethyl sulfoxide and dimethyl formamide. By halogenated hydrocarbons for example methylene chloride or chloroform dissolved in an excess of ammonia the boiling point of the dyeing medium can be advantageously raised.

The following examples serve to illustrate the invention but they are not intended to limit it thereto.

EXAMPLE 1

A fabric of acrylic fibers was padded with a bath containing 5 g/l of the basic dyestuff (U.S. Pat. 3,345,355) of the formula



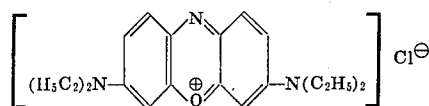
dissolved in anhydrous, liquid ammonia, at -40°C and under atmospheric pressure by means of a suitable airtight unit and squeezed off. The apparatus used consisted of a preparing vessel, a padding foulard, a heatable roller for removing the ammonia, inlet and outlet roller locks and a system for recovering the ammonia with compressor and cooler. When passing the heatable roller, the ammonia was removed from the textile material by evaporation and the goods left the apparatus in dry condition.

The dyestuff applied in this manner was fixed by steaming for 30 minutes under a pressure of 0.2 atmospheres gauge, the goods were rinsed cold and soaped at 95°C with an aqueous dyebath containing 1 g/l of a non-ionic detergent of the type of the reaction product from 1 mol of nonylphenol and 20 mols of ethylene oxide.

There was obtained a brilliant golden yellow dyeing the fastnesses to light and washing of which corresponded to an equally deep dyeing obtained according to the exhaustion method with this dyestuff from an aqueous bath.

EXAMPLE 2

Parallel-run yarns of polycrylic fibers were padded with a dyebath containing 5 g/l of the cationic dyestuff (C.I. 51,004) Basic Blue 3 - C.I. No. 51004 - corresponding to the formula

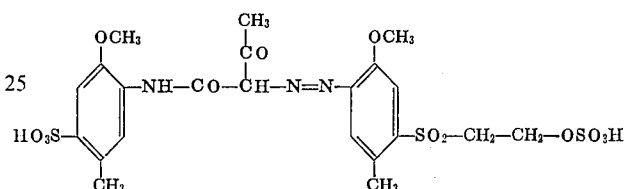


dissolved in anhydrous, liquid ammonia, at -40°C and under atmospheric pressure in the apparatus described in Example 1. The goods were then spooled and the packages were then steamed for 30 minutes at 105°C for fixing the dyestuff. The dyeing was then after-treated in the usual manner and finished.

There was obtained a bright greenish blue dyeing having properties of fastness which corresponded to those of a dyeing obtained according to the exhaustion method from an aqueous medium.

EXAMPLE 3

Piece goods of bleached cotton were padded with a dyebath containing 5 g/l of the reactive dyestuff (U.S. Pat. No. 3,519,616) corresponding to the formula

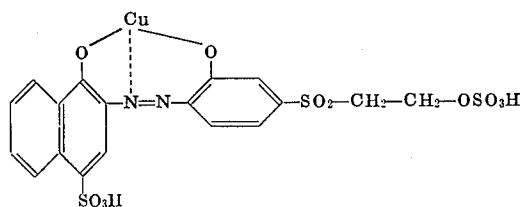


dissolved in liquid ammonia as described in Example 1 and then wound up. The goods were then slop-padded with an aqueous dyebath containing 30 ml/l of sodium hydroxide solution of 32.5 percent strength (38°Be) and 200 g/l of sodium chloride, and the dyestuff was fixed by steaming for 40 seconds at 104°C . The dyed material was then rinsed, acidified and soaped at boiling temperature in an aqueous bath containing 0.5 g/l of a non-ionic detergent of the type of the reaction product from 1 mol of nonylphenyl and 10 mols of ethylene oxide.

There was obtained a brilliant yellow dyeing having the properties which are characteristic for this dyestuff on cotton.

EXAMPLE 4

The dyeing was carried out as described in Example 3 but with piece goods of grey viscose rayon and in a bath containing 10 g/l of the reactive dyestuff (U.S. Pat. No. 3,135,730) of the formula



dissolved in liquid ammonia.

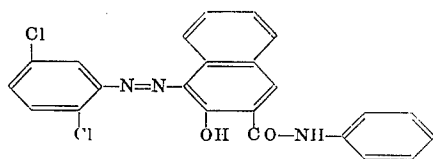
There was obtained a claret dyeing the properties of fastness of which corresponded to those equally deep dyeings obtained with this dyestuff on regenerated cellulose according to the exhaustion method in an aqueous medium.

EXAMPLE 5

Piece goods of linear polyethyleneglycol terephthalate were padded as described in Example 1 with a dye-

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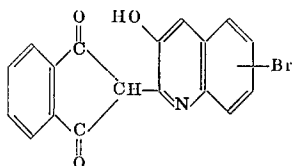
bath containing 5 g/l of the water-insoluble pigment dyestuff (C.I. 12,310) corresponding to the formula



dissolved in anhydrous, liquid ammonia at -45°C , and then wound up. The dyestuff was fixed by thermosoling during 1 minute at 190°C on a thermofixation frame. The material was then subjected to a reductive cleaning and a rinsing process in the usual manner. A red dyeing was obtained.

EXAMPLE 6

Filaments of textured polyester materials were impregnated by spraying with a dyebath containing 1 g/l of the disperse dyestuff (C.I. 47,023) of the formula

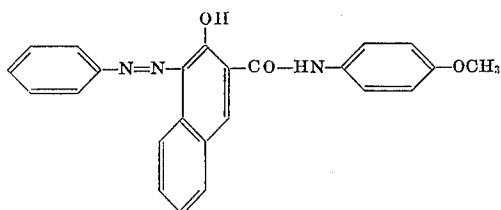


dissolved in liquid ammonia at -40°C . The filaments thus treated were then passed through a heated cylinder. This apparatus was air-tight as the equipment mentioned in Example 1 and provided with an ammonia recovering device. The filaments were then wound off in strands and the dyestuff was fixed for 20 minutes under a pressure of 1.5 atmospheres gage in a high temperature star steamer.

A brilliant light yellow dyeing was obtained. In this case an after-treatment was not necessary.

EXAMPLE 7

Piece goods from a mixture of polyester and cotton at a ratio of 67 : 33 were impregnated as described in Example 1 but in a bath containing 10 g/l of the disperse dyestuff (German Pat. No. 1,131,639) of the formula



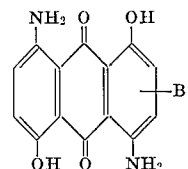
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dissolved in liquid ammonia at -60°C , and then wound up. Fixation of the dyestuff and after-treatment of the dyed goods was carried out as described in Example 5.

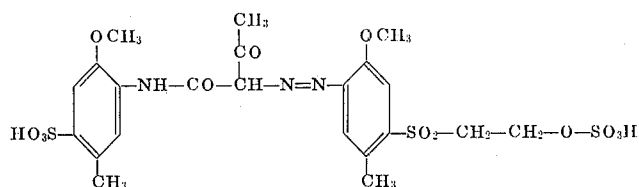
The polyester portion of the mixed fabric was dyed orange tints. The properties of fastness corresponded to those of a dyeing obtained from an aqueous medium.

EXAMPLE 8

A mixed fabric of polyester and cotton in a ratio of 50 : 50 was impregnated as described in Example 1, but with a dyebath containing 5 g/l of the disperse dyestuff (U.S. Pat. No. 2,990,413) of the formula



and 5 g/l of the reactive dyestuff (U.S. Pat. No. 3,519,616) of the formula



dissolved in liquid ammonia at -40°C , and then wound up. The textile material was then treated at 200°C for 45 minutes on a hotflue to fix the disperse dyestuff. In order to fix the reactive dyestuff the goods were then treated for 15 seconds at boiling temperature in a heated trough of the open-width washing machine with an aqueous bath containing 250 g/l of sodium chloride and 50 ml/l of sodium hydroxide solution of 32.5 percent strength (38°Be) and then subjected to the following cleaning operations in the 8 boxes of the open-width washing machine:

1st box: rinsing at 40°C with water

2nd box: acidifying at 40°C with an aqueous bath containing 1 ml/l of acetic acid of 30 percent strength

3rd box: rinsing at 60°C with water

4th box: soaping at 90°C with an aqueous bath containing 0.5 g/l of the reaction product from 1 mol of nonylphenol and 10 mols of ethylene oxide

5th box: soaping at 90°C with an aqueous bath containing 0.5 g/l of the reaction product from 1 mol of nonylphenol and 10 mols of ethylene oxide

6th box: rinsing at 90°C with water

7th box: rinsing at 90°C with water and

8th box: rinsing at 40°C with water.

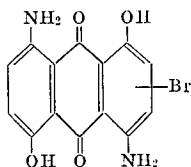
The goods were then dried on a stretching frame.

There was obtained a bi-color dyeing with blue polyester and yellow cotton portion the fastnesses of which corresponded to the fastnesses of a high temperature beam dyeing from an aqueous medium.

EXAMPLE 9

Parallel-running yarns of polyamide 6-fibers were impregnated as described in Example 2 but with a bath

containing 10 g/l of the disperse dyestuff (U.S. Pat. No. 2,990,413) of the formula



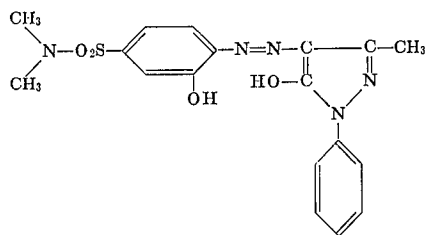
dissolved in liquid ammonia and, after leaving the plant, wound on a warp beam, passed from there over a cylinder heated by means of oil (contact fixation equipment) and re-wound on another warp beam. The dyestuff was fixed thereby. Time of contact: 1 minute, temperature 190°C.

The warp was then interwoven with the weft yarn of viscose rayon staple fiber yarn. The piece goods thus obtained were then subjected on the jig during 4 passages at 60°C to the influence of an aqueous solution of 1 g/l of a non-ionic detergent on the basis of the reaction product from 1 mol of nonylphenol and 10 mols of ethylene oxide and after-treated by repeated rinsing.

The treated fabric had the character of a Denim article with blue polyamide warp and white staple fiber weft yarn.

EXAMPLE 10

Piece goods of polyamide 6-filaments were treated as described in Example 1 with a bath containing 10 g/l of a chromium complex compound of the dyestuff (coupling product of diazotized 2-aminophenol-5-sulfonic acid dimethylamide with 3-methyl-5-hydroxy-pyrazol chromium complex) of the formula



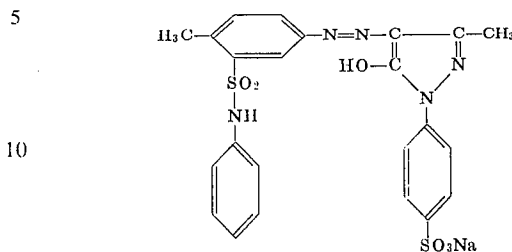
dissolved in liquid ammonia at -50°C, and then steamed for 45 minutes at 104°C in a star steamer. The dyed textile material was then after-treated for 20 minutes at 50°C in a winch vat, first with an aqueous bath containing 1 g/l of an anionic detergent of the type of oleylmethyltaurine and then with a cold aqueous rinsing bath.

There was obtained an even red dyeing the properties of fastness of which did not differ from the fastnesses of a corresponding dyeing obtained from an aqueous medium.

EXAMPLE 11

Yarns of a textured polyamide 6-material were

treated as described in Example 2 with a bath containing 16 g/l of the acid dyestuff Acid Yellow 25 - C. I. No. 18835 corresponding to the formula



dissolved in liquid ammonia at -40°C, and the goods were then steamed for 30 minutes at 104°C in the star steamer. The yarns were then after-treated for 15 minutes at 50°C in a yarn dyeing machine with an aqueous bath containing 0.5 g/l of a non-ionic detergent of the type of the reaction product from 1 mol of nonylphenol and 10 mols of ethylene oxide.

The yarns showed a yellow dyeing the properties of fastness of which did not differ from the properties of an analogous dyeing from an aqueous medium.

We claim:

1. In a process for dyeing textile materials of natural or synthetic fibers alone or in mixture with each other, the improvement which comprises applying to the fibrous material a dyestuff dissolved in a mixture of anhydrous, liquid ammonia and halogenated hydrocarbon selected from the group consisting of methylene chloride, chloroform, and a combination thereof.

2. The improvement defined in claim 1, wherein the dyestuff solution is applied by dipping, padding or spraying.

3. The improvement defined in claim 1, wherein the dyestuff applied is fixed by heat.

4. The improvement defined in claim 1, wherein the dyestuff is a cationic dyestuff.

5. The improvement defined in claim 1, wherein the dyestuff is a dyestuff containing fiber-active groups.

6. The improvement defined in claim 1, wherein the dyestuff is a disperse dyestuff or a water-insoluble organic pigment dyestuff.

7. The improvement defined in claim 1, wherein the dyestuff is an acid dyestuff.

8. The improvement defined in claim 1, wherein the dyestuff is a water-soluble anionic or non-ionic dispersed metal complex dyestuff.

9. The improvement defined in claim 1, wherein textile material is a synthetic polyamide.

10. The improvement defined in claim 1, wherein the textile material is a linear polyester.

11. The improvement defined in claim 1, wherein textile material is polyacrylonitrile.

12. The improvement defined in claim 1, wherein the textile material is natural regenerated cellulose.

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