United States Patent [19]

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[11] **3,929,407**

[45] **Dec. 30, 1975**

[54] **PROCESS FOR DYEING OF NOVOLOID FIBERS**

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[22] Filed: **June 24, 1974**

[21] Appl. No.: **482,307**

[52] **U.S. Cl.** ..... **8/168;** 8/DIG. 3; 8/73; 8/92; 8/115.5; 8/173; 8/176; 264/176 F

[51] **Int. Cl.²** **C08J 3/20**

[58] **Field of Search** ..... 8/DIG. 3, 73, 168, 176, 8/115.5, 92, 173; 264/176 F

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[57] **ABSTRACT**

A process for dyeing novoloid fiber without changing the thermal properties or altering the hand (textural quality or feel). After scouring and rinsing, the fabric is placed in an enclosed container with water, 1 – 5 grams per liter of a carrier such as Butyl benzoate and 0.10 – 10.0 per cent of a basic dye, such as Victoria Green Crystals. The pH in the container is maintained at approximately 4.5 to 6.5 with acetic acid or another suitable chemical. The temperature is raised at an approximate rate of 2 Farenheit degrees per minute from 100°F to 240° – 300°F and then held for at least 1 ½ hours at that temperature. The container is cooled to 140°F – 180°F, rinsed until clear and the fabric removed. Finally, the fully relaxed fabric is extracted and framed dryed at 250°F – 350°F.

14 Claims, No Drawings

PROCESS FOR DYEING OF NOVOLOID FIBERS

BACKGROUND OF THE INVENTION

This invention relates generally to the field of bleaching and dyeing of textiles and fabrics and more particularly to a process for dyeing novoloid fibers.

Novoloid fibers are generating increasing amounts of interest for use as textile material for certain types of protective clothing because of their superior flame retardant and heat resistant properties. One of the principal reasons for these desirable properties is the unique cross-linked phenolic molecular structure of the novoloid fiber. When exposed to temperatures as high as 4,500°F the fiber will neither melt nor fuse, but will turn to carbonized fiber. Upon exposure to extreme heat, fabrics produced from these fibers will release noncombustible, nontoxic gases, while retaining their shape. Novoloid fibers therefore most logically offer a promising outlook for further improvement of thermal protection over presently used fiber materials, such as polybenzylimidazoles (PBI) and aromatic polyamides (Nomex).

One of the more difficult problems encountered however, has been the poor dye acceptance of such fibers because of the lack of dyesites on the novoloid fiber molecule for accepting a dyestuff. Under normal conditions the novoloid fiber does not react with complete classes of dyestuffs. By means of the present invention, selective chemical compounds (dyestuffs) are chemically attached to the molecular structure of the novoloid fiber.

Numerous requirements exist for protective clothing of a particular color. One such requirement is the clothing worn by certain personnel on the flight deck of an aircraft carrier, which, by regulation, must be bright green or orange according to their particular jobs in aircraft direction and handling. Another example is the bright orange flight suits worn by Navy pilots for rescue purposes in the event that they should become separated from their aircraft. Several other examples include parachute covers and survival vests.

SUMMARY OF THE INVENTION

Accordingly, it is a general purpose and object of the present invention to provide a process for dyeing novoloid fiber, yarn or fabric without changing the thermal properties or altering the hand. It is another object to provide a process for dyeing novoloid fiber with selected cationic and dispersed dyestuffs.

These and other objects are accomplished according to the present invention by a process including the following steps:

a placing the fabric in an approximately 100°F mixture of (1) water,

(2) 1–5 grams per liter of Butyl benzoate carrier, (3) 0.10 percent – 10 percent by weight of fabric of a cationic dyestuff, (4) a sufficient quantity of a suitable chemical for maintaining a pH of essentially 4.5 to 6.5;

(b) heating the mixture to a high temperature of approximately 240°F – 300°F and holding at that temperature for at least 1 ½hours;

(c) cooling the mixture and fabric to approximately 140°–180°F.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Novoloid fiber is defined as a manufactured fiber containing at least 85 percent by weight of a cross-linked novolac. The composition of the fiber may be considered with reference to the basic aspects of the method by which such fibers may be made. A phenolicaldehyde novolac is prepared by condensing a phenolic compound and an aldehyde in the presence of an acid catalyst, a stochiometric excess of the phenolic compound preferably and customarily being employed. The novolac so obtained is fiberized by any conventional method such as melt spinning. The resulting novolac fibers are then heated in an environment containing formaldehyde and an acid catalyst to effect curing, (i.e., cross-linking of the novolac). Such cross-linking occurs primarily by virtue of methylene bridges formed between the 2, 4 and/or 6 positions of the phenolic structural unit. The terms "phenolic compound" in the present context refers primarily to the compound phenol itself, from which most novolacs are prepared. However, it also encompasses phenol wherein one or more of the hydrogen atoms are substituted by a monovalent radical, provided, however, that the phenol is not so extensively substituted in the 2, 4 and/or 6 positions as to preclude subsequent cross-linking. Cresol is a primary example of such a substituted phenol. Similarly, the term "aldehyde" refers to any aldehyde capable of condensing with the phenolic compound to form a novolac. Formaldehyde is by far the most commonly used, but other aldehydes such as acid aldehyde may be employed. The novolac melt from which the instant fibers are formed preferably contains at least 50 percent of a novolac produced from phenol and formaldehyde.

The novoloid fiber so produced has a natural rust-/gold color and is most significantly characterized by remarkable resistance to heat and flame, being infusible and nonflamable. It is also substantially unaffected by many acids, and is insoluble in inorganic solvents.

The present invention will now be more fully described by reference to the following examples.

EXAMPLE 1

Approximately 60 pounds of novoloid fiber staple was subjected to the following process in order to produce novoloid yarn:

a the novoloid staple was put through a Picker twice in order to open up the fibers;

b a woolen card was used in the carding process to make enough novoloid sliver for the spinning frame (worsted and cotton cards were not successful in producing the sliver);

c the slivers were drawn out with two passes before being placed into the spinning process;

d A Saco Lowell cotton spinning frame was used to convert the slivers into 36/1 novoloid yarn (it was necessary to spray the yarn during the process with isopropyl alcohol to insure continuous runability);

e the 36/1 novoloid yarn was finally plied to a cotton count of 36/2 before the warping and knitting or weaving steps. The above process describes but one method for producing a particular yarn size. It is to be understood that any other suitable process for producing other yarn sizes could be substituted without altering the present invention.

An RM6-36 gauge Mayer Raschel knitting machine, for example, was used to knit the novoloid yarn into a fabric after experimentation with various stitch formations to arrive at the best possible fabric construction. The following specifications apply to the 100 percent novoloid knit fabric produced;

Yarn:	36/2 100% Novoloid
Stitch Formation:	Front Bar 2/0 4/6 Back Bar 6/6 0/0
Fabric Width:	42 inches 26" Quality
Greige Weight:	7.33 Ounces/yard 2
Finished Weight:	7.0 Ounces/yard 2
Construction:	Wales — 18 Courses — 20
Stoll Abrasion 2 lb Load - 3 P.S.I.: "0" Emery Cloth	205
Air Permeability: 0.5" H_2O Pressure:	208 - cu. ft. OF Air per sq. ft. OF fabric per minute

A sample of this novoloid knit fabric was then dyed in a pressurized dyeing machine, such as a package dyer or beam dyeing machine. Various dyestuffs, including cationic (basic) and dispersed dyestuffs were selected by the following criteria — ease of solubility, high solubility and shade desired. The process used to dye the first knit novoloid fabric sample was as follows: (a) the fabric is scoured and rinsed by immersing it in water with approximately 2–3 grams per liter of a detergent such as an ethylene oxide condensate with a sodium salt of a fatty acid detergent such as (Allo Scour TY). The bath temperature is maintained at 180°F. The fabric is agitated or the scouring solution is pumped through the fabric. Thorough rinsing with clear water is necessary to completely remove the scouring solution. (b) the fabric is placed in an approximately 100°F mixture of, (1) water (2) 1–5 grams per liter of a typical carrier of the DAC – 888 type known as Butyl benzoate, used to assist the dye in penetrating the fiber, (3) 0.1 to 10.0 percent by weight of the fabric of a cationic dyestuff, Victoria Green Crystals (Basic Green 4), (4) sufficient acetic acid CH_3COOH, or other suitable chemical to hold the pH at approximately 4.5–6.5; Examples of other chemicals which could be used to control the pH are sodium citrate, mono sodium phosphate, mono potassium phosphate, acetic acid - sodium acetate and hydrogen sulfide.

c. the temperature of the mixture is raised at an approximate rate of 2 Fahrenheit degrees per minute to a high temperature of 240°0 – 300°F and held at the elevated temperature for at least 1 ½ hours;

(d) the mixture and fabric are then cooled to approximately 140°F – 180°F, rinsed until clear, and the fabric removed;

(e) the fully relaxed fabric is then extracted and frame dryed at 250° – 350°F in a chamber.

EXAMPLE 2

The above process was repeated using a cationic dyestuff, Sevron Green B (Basic Green 3).

EXAMPLE 3

The Above process was repeated using a dispersed dyestuff, Palanil Violet 3R (Dispersed Violet 8).

EXAMPLE 4

The above process was repeated using a dispersed dyestuff, Palanil Yellow 3G (Dispersed Yellow 64).

EXAMPLE 5

The above process was repeated using a dispersed dyestuff, Polydye Burnt Orange 0 (Dispersed Orange 59).

EXAMPLE 6

The above process was repeated using a dispersed dyestuff, Latyl Brilliant Blue BLT (Experimental).

EXAMPLE 7

The above process was repeated using a dispersed dyestuff, Palanil Black (Mixture).

EXAMPLE 8

The above process was repeated using a dispersed dyestuff, Palanil Blue R (Dispersed Blue 56).

EXAMPLE 9

The above process was repeated using a dispersed dyestuff, Palanil Brilliant Pink REL (Dispersed Red 91).

EXAMPLE 10

The above process was repeated using a dispersed dyestuff, Palanil Blue RT (Mixture).

The results of tests made upon the dyed novoloid fabrics were within acceptable limits for color fastness and observation of the hand. The previously described outstanding thermal properties of the undyed fabric were found not to have been altered by the dyeing process. Thus, novoloid fibers and fabrics having the advantages of considerable flame retardant and heat resistant properties can now be used for clothing which is required to be dyed a specific color.

Having thus described the present invention, some of the advantages thereof should now be readily apparent. The dyed novoloid fabric present a superior heat and flame resistant fabric useable, for example, as clothing which must be a specific color for personnel working in dangerous heat and flame environments.

This invention has been described in connection with certain specific embodiments thereof. However, it should be understood that these are by way of example rather than by way of limitation, and it is not intended that the invention be restricted thereby.

What is claimed is:

1. A process for dyeing an unmodified novoloid fiber comprising the steps of:

a. placing the fiber in an approximately 100°F mixture of:

1 water,

2 1 to 5 grams per liter of butyl benzoate dyestuff carrier,

3 0.1 to 10.0 per cent based on weight of fiber of the group consisting of cationic and dispersed dyestuffs,

4 a sufficient quantity of a suitable chemical for maintaining a pH of the mixture of essentially 4.5 to 6.5;

b. heating the mixture to a high temperature of between 240°F to 300°F;

c. holding the mixture at the high temperature of step (b) for at least 1 ½ hours; and

d. cooling the mixture and fabric to approximately 140°F to 180°F.

2. A process as set forth in claim **1** wherein the chemical of step (a) (4) is acetic acid.

3. A process as set forth in claim 1 wherein the dyestuff is cationic.

4. A process as set forth in claim 3 wherein the cationic dyestuff is Basic Green 4.

5. A process as set forth in claim 3 wherein the cationic dyestuff is Basic Green 3.

6. A process as set forth in claim 1 wherein the dyestuff is dispersed.

7. A process as set forth in claim 6 wherein the dispersed dyestuff is Dispersed Violet 8.

8. A process as set forth in claim 6 wherein the dispersed dyestuff is Dispersed Yellow 64.

9. A process as set forth in claim 6 wherein the dispersed dyestuff is Dispersed Orange 59.

10. A process as set forth in claim 6 wherein the dispersed dyestuff is Latyl Brilliant Blue BLT.

11. A process as set forth in claim 6 wherein the dispersed dyestuff is Palanil Black.

12. A process as set forth in claim 6 wherein the dispersed dyestuff is Dispersed Blue 56.

13. A process as set forth in claim 6 wherein the dispersed dyestuff is Dispersed Red 91.

14. A process as set forth in claim 6 wherein the dispersed dyestuff is Palanil Blue RT.

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