FLAME RETARDANT FOR TEXTILES
5 Claims, No Drawings

U.S. Cl. .............................................. 117/137, 117/143 A

Int. Cl. ........................................ C09k 3/28,
D06m 13/26

Field of Search ................................ 117/136,
143, 217; 8/116, 116.2; 252/8.1; 106/15 FP

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ABSTRACT: Aqueous solutions of dimethylol dicyandiamide
and phosphoric acid useful as durable finishes on cellulose
textile materials to impart flame retardance and the cellulosic
textile materials treated therewith.
3,625,753

1 FLAME RETARDANT FOR TEXTILES

This invention relates to an aqueous flame-retardant finish for cellulosic textile materials. More particularly, it relates to (1) aqueous solutions of dimethyl dicyandiamide and phosphoric acid, (2) to their use as a durable finish on cellulosic textile materials to impart flame retardancy, and (3) to the treated cellulosic textile materials.

The use of monomethyl dicyandiamide in flame-retardant finishes for cellulosic textile materials is well known. For example, U.S. Pat. No. 2,519,388, discloses its use in combination with phosphoric acid and methylol urea or a methylol melamine. The coapplication with an aminoplast resin, such as a methylol melamine, is necessary to obtain good wash durability of the flame-retardant finish. Also, monomethyl dicyandiamide is costly to manufacture. Because of insolubility in water, monomethyl dicyandiamide must be isolated and the isolation steps add considerably to the cost of the product, thus resulting in a price deterrent to the customer.

It is therefore an object of the invention to provide a flame-retardant finish for cellulosic textile materials which is economical and durable to laundering.

This and other objects of the invention will become apparent as the description thereof proceeds.

It has now been discovered that a dimethyl dicyandiamide and phosphoric acid flame-retardant system does not require the coapplication of an aminoplast resin, such as a methylol melamine, in order to obtain durability of the finish on cellulosic textile materials. Furthermore, dimethyl dicyandiamide is water-soluble and can be manufactured and used in the flame-retardant finish with isolation. An added advantage of the flame-retardant finish of this invention is the relatively small strength loss sustained by the textile materials when reacted with the finish.

The dimethyl dicyandiamide can be prepared by heating a mixture of dicyandiamide with two-mole equivalents of aqueous formaldehyde (formalin) to a temperature of about 70°C. Heating of the clear solution is continued for about 1 hour. The resulting aqueous solution of dimethyl dicyandiamide contains 50–60 percent solids and it can be used in the process of this invention.

The phosphoric acid used in the process of this invention is preferably orthophosphoric acid (H₃PO₄). The commercial 85 percent acid is conveniently employed.

The dimethyl dicyandiamide-phosphoric acid finish can be conveniently applied to textile materials as an aqueous solution. The textile material can be treated by padding, spraying, dipping etc. The cellulosic textile material should contain cellulose fibers by the term cellulose fibers as the term "cellulose fibers" is meant such fibers as cotton, regenerated cellulose (rayon), linen, jute, etc. Blends of cellulose fibers with other cellulose fibers or with noncellulose fibers, both natural and synthetic, such as silk, wool, polyamide, polysteres, polyacrylonitrile, e.g., may be used.

The amount of the dimethyl dicyandiamide-phosphoric acid finish applied to the textile material should be between 30 and 45 percent based on the weight of the textile material. The amount of dimethyl dicyandiamide in the finish should be between 15 and 40 percent, preferably between 20 and 30 percent, based on the weight of the textile material, and the amount of phosphoric acid should be between 5 and 20 percent, preferably between 7.5 and 15 percent, based on the weight of the textile material. Furthermore, the amount of dimethyl dicyandiamide and phosphoric acid in the aqueous solution will be adjusted to provide the amount of pickup on the textile material, as pointed out above, and will depend on the method of application. It is within the skill of the art to determine the amounts required in the solution.

The treated textile material is dried by any convenient means, as in an oven at from about 200°C to 250°C, preferably 225°C, and is then heated at an elevated temperature to effect a "cure" of the finish on the textile material. A temperature between 225°C and 400°C, preferably between 250°C and 350°C, is recommended for the curing operation. This time required is between about 1 and 5 minutes depending on the temperature used and the weight of the fabric. If desired, the drying and curing operation can be done in one step.

The following specific examples are set forth to illustrate some embodiments of the invention and are not intended to be limiting.

EXAMPLE I

Preparation of Dimethyl Dicyandiamide

A mixture of 1,180 g. (14 moles) dicyandiamide and 2,330 g. of 35 percent aqueous formaldehyde (28 moles) was heated slowly to 70°C, whereupon the mixture became clear. Heating was then continued at 75°C for one hour. The product contained about 55 percent solids.

EXAMPLES II–XXIV

Twenty-three aqueous pad baths were prepared containing the percentage amounts on a solid basis of dimethyl dicyandiamide (DD), product of example 1, and orthophosphoric acid (H₃PO₄) shown in Table I.

The pad baths were applied to 80×80 percale by a standard padding procedure obtaining a 82 percent wet pickup. The padded fabrics were dried at 225°F. for 2 minutes and then heated at 325°F. for 3 minutes.

The durability of the fire-retardant finish to laundering was determined by repeatedly washing the fabric in an automatic washing machine using water at 140°F. and a commercial detergent. After each washing operation, the flame resistance of the fabrics was determined by a vertical flame test according to Standard Test Method AATCC 34-1966. The washings and flame tests were continued as long as the char length of the burnt fabric was less than 6 inches. The limit of wash durability was reached when the char length was 6 inches.

The amounts of dimethyl dicyandiamide and phosphoric acid on the textile material and the durability of each finish to laundering are shown in Table I.

| TABLE I |
| Pad bath composition, percent | Treated textile material, percent OWF | Durability of finish |
| Example number | DD | H₃PO₄ | DD | H₃PO₄ | No. of washes |
| 2 | 15 | 2.0 | 12.5 | 2.1 | 0 |
| 3 | 15 | 6.1 | 12.5 | 2.1 | 1 |
| 4 | 15 | 7.8 | 12.5 | 2.1 | 3 |
| 5 | 15 | 10.9 | 12.5 | 2.1 | 8 |
| 6 | 20 | 3.4 | 18.5 | 2.0 | 0 |
| 7 | 20 | 6.5 | 18.5 | 2.0 | 1 |
| 8 | 20 | 10.2 | 18.5 | 2.0 | 4 |
| 9 | 20 | 13.6 | 18.5 | 2.0 | 9 |
| 10 | 25 | 4.2 | 22.0 | 2.5 | 2 |
| 11 | 25 | 9.5 | 22.0 | 2.5 | 9 |
| 12 | 25 | 12.7 | 22.0 | 2.5 | 11 |
| 13 | 25 | 17.0 | 22.0 | 2.5 | 9 |
| 14 | 25 | 8.1 | 24.6 | 4.2 | 8 |
| 15 | 25 | 10.2 | 24.6 | 4.2 | 13 |
| 16 | 25 | 13.3 | 24.6 | 4.2 | 11 |
| 17 | 35 | 20.4 | 24.6 | 10.7 | 7 |
| 18 | 35 | 11.9 | 28.7 | 0.9 | 17 |
| 19 | 35 | 17.8 | 28.7 | 14.9 | 5 |
| 20 | 35 | 25.8 | 28.7 | 10.5 | 4 |
| 21 | 40 | 6.8 | 22.6 | 0.6 | 16 |
| 22 | 40 | 13.9 | 22.6 | 11.1 | 19 |
| 23 | 40 | 20.4 | 22.6 | 15.7 | 11 |

We claim:

1. A process for producing a durable flame-retardant finish on cellulosic textile materials comprising the steps of:
   a. forming a coated cellulosic textile by applying a flame-retardant composition consisting essentially of an aqueous solution of dimethyl dicyandiamide and phosphoric acid to said cellulosic textile materials, wherein the amount of dimethyl dicyandiamide and phosphoric acid are in the ranges of 15 to 40 percent and 5 to 20 percent, respectively, based on the weight of the textile; and
   b. heating the coated textile to dry and cure the finish thereon.
2. The process of claim 1 wherein said coated textile is subsequently heated to a temperature from about 225° to 400° F.

3. The process of claim 2 wherein said temperature is from about 250° to 350° F.

4. The process of claim 1 wherein said textile is dried at a temperature of from about 200° to 250° F. and subsequently heated to from 250° to 350° F. to cure said finish.

5. A cellulosic textile material obtained by the process of claim 1.
UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,625,753 Dated December 7, 1971

Inventor(s) Samuel James O'Brien and Robert George Weyker

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 1, line 32, "with" should read -- without --.

Column 1, line 36, "dicyandimide" should read -- dicyandiamide --.

Column 1, line 75, "This" should read -- The --.

Signed and sealed this 9th day of May 1972.

(SEAL)
Attest:

EDWARD M. FLETCHER, JR. ROBERT GOTTSCHALK
Attesting Officer Commissioner of Patents