United States Patent Office 3,450,484
Patented June 17, 1969

3,450,484

METHOD OF PRODUCING FABRICS FOR CREASE-RETAINING GARMENTS

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No Drawing. Filed Aug. 9, 1965, Ser. No. 478,417
Int. Cl. D06m 13/14
U.S. Cl. 8—116.3 3 Claims

ABSTRACT OF THE DISCLOSURE

This specification discloses a process for producing a cellulose containing fabric capable of having permanent creases made therein by heat and pressure, by impregnating the fabric with an aqueous dispersion of a condensation product of a lower alkyl carbamate and formaldehyde, and drying the impregnated fabric at elevated temperatures to the point where the fabric contains 4.75 to 3.0% moisture. This moisture level is actually below the equilibrium moisture content of cotton under typical ambient conditions.

This invention relates to the production of crease-resistant fabrics of the so-called deferred cure type, in which final cure of the crease-resisting fabric finish is delayed until after articles are made from the fabric, so that permanent creases can be made in the articles, and has as its principal object the elimination of the odor problem which has heretofore arisen with these materials.

Crease-proofing of cellulosic fabrics is an old art. Many years ago, it was discovered that if such a fabric was impregnated with a water-soluble thermosetting resin, and the resin cured on the fabric, a finish was obtained which gave materials which were resistant to creasing. Originally, the useful finishes were the heat-convertible resins, made by heating amino compounds like urea, melamine, biuret, ammeline, etc., with formaldehyde. More recently, it was discovered that cellulose itself would react with certain finishing agents which were not in themselves resins, and would not form resins on simply heating alone. Of these materials the condensation products of the lower alkyl carbamates and formaldehyde have proven the most promising, in particular the condensates of formaldehyde with methyl carbamate and ethyl carbamate.

In the development of these crease-resistant fabrics, a recent commercial development is the so-called deferred cure process. In this process, fabric is impregnated with the crease-resistant finish, and the fabric is dried very carefully to prevent premature cross-linking of the finish. Cross-linking of the finish is deferred until after the fabric is cut up and made into articles such as garments; the desired creases are pressed into the garments, the heat of the pressing irons acting to further cross-linking, with final cure of the finish coming after pressing. The method has been suggested for both amino-formaldehyde resin finishes (see Warnock et al., U.S. Patent 2,974,432, issued Mar. 14, 1961) and for cellulose-reactive finishes of the alkyl carbamate-formaldehyde type (see Kullman et al., American Dyestuff Reporter, Mar. 2, 1964, pages 22 and 23).

While the deferred cure process is technically sound, it has one very important disadvantage. Both the amino-formaldehyde resin finishes and the alkyl carbamate-formaldehyde finishes contain loosely bound formaldehyde which splits off during the final curing, and makes working with these finishes most unpleasant in the absence of good ventilation—a convenience notably lacking in many lofts in which garments are finished. Deodorants

have therefore been widely used in the formation of deferred cure finishes. However, this adds to the cost, and ends up by merely masking the problem as well as the odor. In many shops, the "deodorized" finish odors prove just as objectionable as formaldehyde.

We have discovered that the odor problem can be eliminated in deferred cure processing of cellulosic fabrics with alkyl carbamate-formaldehyde finishes, by the very simple expedient of drying the fabric to a moisture content of from about 4.75 to about 3%. This is actually below the equilibrium moisture content of cotton under typical ambient conditions, e.g., 5.0% at 75° F. and 40% relative humidity. Surprisingly, it appears at this point that just sufficient cross-linking has occurred to fix any unvolatilized formaldehyde to the cellulose without going to the point where the desired advantages of conventional fully deferred curing are lost. This is directly contrary to the teachings of prior art investigators who have always retained a substantial amount of moisture in their fabrics, in order to prevent substantial cross-linking before processing into clothing.

It is even more surprising that our new method is applicable in the practice of the invention taught in our copending application Ser. No. 368,362, filed May 18, 1964. This application teaches the use of a pH range of about 2 to 4 in the fabric treating bath, which accelerates cure and produces a fabric of improved strength because of the milder conditions to which the fabric has to be subjected during the cure.

In the practice of our invention, we may use any of the lower alkyl carbamates, including methyl, ethyl, n-propyl, isopropyl, and sec-butyl carbamates. The methyl, ethyl, and isopropyl carbamates are preferred.

Alkyl carbamates are commercially available products which may be prepared in various ways which are well known to those skilled in the art. Typical processes for preparing alkyl carbamates are described in U.S. Patents 3,013,065 and 3,013,064. These carbamates are generally prepared by dissolving urea in the corresponding alcohol up to the solubility limit of urea in alcohol. Alkyl carbamates may also be prepared by the reaction of ammonia and an alkyl chloroformate.

The alkyl carbamate-formaldehyde condensates used herein may be prepared in accordance with well-known techniques by reacting 2–3 moles of formaldehyde with each mole of alkyl carbamate in aqueous medium at a pH of 2–10.5. The pH is controlled by the addition of a basic material such as sodium hydroxide, sodium carbonate, barium hydroxide, triethanolamine, etc. The reaction will take place by standing overnight at room temperature, or by mildly heating for a shorter period of time such as 45° C. for two hours. The reaction initially takes place to the extent of condensing about 1.5–1.6 moles of formaldehyde with each mole of carbamate and the excess formaldehyde remains dissolved in the aqueous medium. When the reaction equilibrium is reached, the condensate is stabilized by lowering the pH to about 7 by the addition of acid such as hydrochloric or acetic. The specific method by which the condensate is prepared is not critical to the success of this invention.

The impregnating solution, which is often called the "pad bath," is prepared by adjusting the concentration of the active material (i.e. the carbamate-formaldehyde condensate) in the aqueous medium to about 2–30%, and preferably about 8–12%. Magnesium chloride hexahydrate or other catalyst is then added in a concentration which may vary from about 10–100% by weight, based on the condensate, and preferably about 25–40%.

The impregnating solution may be used as such, but it preferably is adjusted to a pH of about 2 to 4, and preferably about 2.5 to 3.5, as taught in our copending application Ser. No. 368,362, filed May 18, 1964. As indicated

there, substantially any organic acid may be used for the adjustment.

Ordinarily there is added to the impregnating solution a conventional softener—e.g., dispersed low molecular weight polyethylene, various fatty acid derivatives including quaternary ammonium and amide derivatives—which acts to give the fabric a soft, pleasant feel. Where desired, a hand builder is also often added, to give body and firmness; typical hand builders are polyvinyl acetate and modified starches.

Any type of cellulose textile material may be rendered wrinkle-resistant in accordance with this invention. Suitable textile materials include natural cellulose products such as cotton, linen, hemp, flax, ramie, sisal, jute, straw, and wood; regenerated cellulose products such as viscose, cuprammonium, and high tenacity-modified viscose rayons. The cellulose textile may be in the form of a fiber, filament, or yarn or it may be in the form of a paper or cloth made by weaving, felting, knitting, knotting, bonding, or crocheting. The cellulose material may also be in admixture with other natural or synthetic fibrous materials, e.g., the conventional 65 polyester fiber:35 cotton blend used in many fabrics.

The impregnated textile, after being impregnated with the desired amount of finishing agent, is now dried, in accordance with this invention, to remove the free moisture to between about 4.75 and 3%, i.e., below the normal equilibrium content of free moisture at ambient conditions. This requires a time-temperature cycle which starts the cross-linking and eliminates not only the free water but all the formaldehyde which will come off. Temperatures of from about 95 to 120° C. are preferred, with times of three to ten minutes, the lower temperatures requiring the longer times. Temperature of the order of as high as 150° C. for a few seconds can also be used, but the resultant moisture content gets more difficult to control as the temperature goes up. Strangely enough, the presence or absence of organic acid in the impregnating solution has little or no effect on the permissible moisture content; it appears that this acid produces most or all of its effect in the final stages of the cure.

The cellulose textile fabrics prepared in this manner can be stored as needed for considerable periods of time, then made into garments or other objects, and after-cured after the desired creases are pressed into the garments, as is conventional in the deferred cure process. However, with the process of this invention, no substantial formaldehyde odor is developed in after-curing, so that the textiles may be conveniently used under any conditions of ventilation.

The following typical examples of the invention are given by way of illustration and not by way of limitation; they can be multiplied indefinitely without departing from the scope of the invention as defined in the claims. In the examples, all parts are by weight.

Example 1.—Preparation of ethyl carbamate-formaldehyde condensate

Eighty-nine parts of ethyl carbamate and 202 parts of 37% formaldehyde (methanol-free) (molar ratio of carbamate to formaldehyde 1:2.5) were charged into a vessel equipped with a stirrer, thermometer, and heating and cooling coils. The pH was adjusted to 10.5±0.1 with 50% NaOH. The exothermic reaction raised the temperature to 35–40° C. within an hour; thereafter the mix was maintained at 38–42° C. for about an additional hour, until the free formaldehyde level dropped to and stabilized at 8–10%, as determined by the method disclosed in Formaldehyde, 2nd ed., J. Frederick Walker, 1953, Reinhold Publishing Co., N.Y., page 383. At this point, the solution was cooled to 25° C., the pH adjusted to 6.9–7.1 with conc. HCl, and then adjusted to 50% active by the addition of 37 parts water.

Example 2.—Preparation of methyl carbamate-formaldehyde condensate

Example 1 was repeated, using 75 parts of methyl carbamate, 202 parts 37% formaldehyde, and 21 parts water at the end to get a 50% active mix.

Examples 3, 4, 5, and 6.—Pad baths

Pad baths were made as follows:

Example	3	4	5	6
Product of Example 1 (lbs.)	80	80		
Product of Example 2 (lbs.)			80	80
50% solution of $MgCl_2$–$6H_2O$ (lbs.)	24	24	24	24
30% solution of maleic acid, to pH 4 (cc.)	ca 300		ca 300	
Softener—25% aqueous dispersion of low molecular weight polyethylene (lbs.)	8	8	8	8

Each of the examples then was made up 400 pounds with water to give 10% concentration of active material.

Each of Examples 3, 4, 5, and 6 was used to impregnate 80 x 80 print cloth (3.75 square yards per pound) and 136 x 64 broadcloth (2.8 square yards per pound) controlling the impregnation for 80% pickup based on fabric weight. Samples of the cloths were then dried to moisture contents of 4.9%, 4.0%, 3.0%, and 2.5% at 105–110° C. Creases were then pressed into the samples by pressing with a hand iron at the "cotton" setting (350–400° F.) with some additional pressure above the weight of the iron, and the pressed samples were then further processed at 320° F. in a circulating air oven (2 minutes for Examples 3 and 5, 4 minutes for Examples 4 and 6). Other samples were pressed on a hot head press operating at about 310–315° F. under 80 p.s.i.g. steam pressure, and then post-cured for 2 minutes at 320° F.

All of the cloth samples which retained 4.9% moisture developed a small but still noticeable formaldehyde odor on pressing, whereas the other samples had no odor on pressing. The samples were then given five home-style launderings; all of them retained their creases acceptably except the 2.5% samples, which showed a marked loss of crease.

Further extensive testing was done on the product of Example 3. Storage stability on the cloths after drying to 4% moisture was determined by prolonged storage at 70–75° F. and 40–65% relative humidity. Samples stored sixteen weeks still produced results equivalent to freshly prepared samples, indicating satisfactory long-range stability.

Both impregnated cloths were then tested under varying pressing and curing conditions, each being pressed for 30 and 60 seconds, and cured for 2 or 3 minutes; this made a total of four combinations of conditions for each of the two cloths. Replicated samples were then given twenty home-style launderings using ordinary hypochlorite bleach. Observations were made after 5, 10, 15, and 20 launderings. The samples all showed essentially no loss of crease, and the flat portions of the laundered cloths were remarkably smooth. Moreover, no damage from the hypochlorite was observed.

Small laboratory runs made with isopropyl carbamate and sec-butyl carbamate gave essential similar results. Moreover, changes in the carbamate formaldehyde molar ratio over the range 1:2 to 1:3 indicated this entire range was useful. Changes were also made in concentrations of active agent in the pad liquor, and results were essentially what would be expected from the smaller or larger concentrations of finishing agent present.

We claim:

1. In the process of producing a cellulose containing fabric capable of having permanent creases made therein by heat and pressure, by impregnating the fabric with an aqueous dispersion containing an operative amount of a condensation product of 1 mol of a lower alkyl carbamate and from 2 to 3 mols of formaldehyde, and dry-

ing the fabric, the improvement which comprises drying the fabric, at temperatures of from 95 to 150° C., to the point where the fabric contains from 4.75 to 3.0% moisture.

2. The process of claim 1, in which the alkyl carbamate is ethyl carbamate.

3. The method of producing permanent creases in a structure made from a cellulose containing fabric which comprises producing a cellulose containing fabric according to the process of claim 1, thereafter working up the fabric into a structure, ironing creases into the fabric by

heat and pressure, and completing the setting of the condensation product by further heating.

References Cited

UNITED STATES PATENTS

2,974,432 3/1961 Warnock et al. ------ 8—116.3

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