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[54] **DELAYED CURE PROCESS USING FORMALDEHYDE VAPOR TO CAUSE CREASEPROOFING**

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[52] **U.S. Cl.** **8/116.4,** 8/115.7, 8/DIG. 21, 8/DIG. 4, 8/DIG. 10, 8/129, 8/116.3, 8/116.2, 38/144, 2/243, 8/149.2

[51] **Int. Cl.** **D06m 1/16,** D06m 13/14, D06m 13/54

[58] **Field of Search** 8/116.4, 116.3, 116.2; 38/144; 2/243

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[57] **ABSTRACT**

Cellulosic materials such as cotton fabrics, preferably preimpregnated with an amide such as urea, are treated with formaldehyde vapor, formed into garments and cured in the presence of a catalyst to impart durable press and wrinkle recovery characteristics to such garments. The required curing catalyst may be furnished either by impregnating the fabrics with an aqueous solution of a weakly acid salt such as zinc chloride after exposure of the fabric to formaldehyde and before garment fabrication therefrom, or by furnishing a gaseous catalyst such as sulfur dioxide to the chamber in which the garments fabricated from the formaldehyde-treated fabric are cured.

12 Claims, No Drawings

DELAYED CURE PROCESS USING FORMALDEHYDE VAPOR TO CAUSE CREASEPROOFING

BACKGROUND OF INVENTION

There is an ever growing demand for durable press garments made from cellulosic fabrics and a need to provide improvements in the production of such garments. For instance, some such known durable press treatments produce skin irritations either when garment workers handle the treated or partially treated fabrics or when garments made from such fabrics are worn. Many durable press treatments also result in sensitized fabrics which have limited storage life after application of creaseproofing agent, and cause substantial losses in tensile strength, abrasion resistance or other desirable original fabric properties. Still other processes are difficult to control and therefore produce widely different results at different times. Moreover, especially in the case of delayed cure processes, they depend on the deposition of reactive, water soluble resins which preclude washing of the fabric between resin application and final cure and which also in other respects make such partially treated fabrics difficult to handle.

Accordingly, it is an object of this invention to provide an improved treatment whereby the aforementioned disadvantages are minimized or avoided. A more specific object is to provide a process for creaseproofing cotton or other cellulosic materials using formaldehyde vapor as a source from which a reactive but water insoluble creaseproofing agent is evenly formed on the fabric in situ and which permits the cure of the treated material to be safely delayed for long or indefinite periods until after completion of desired articles therefrom including, for instance, the cutting, sewing and pressing of garments. Another specific object is to provide a delayed cure process which produces good wrinkle recovery and smooth drying characteristics, keeps loss of tensile strength and abrasion resistance to a minimum, gives good laundering life and chlorine resistance, employs simple monomeric reactants and, if desired, can produce a pretreated fabric which is relatively insensitive to surrounding conditions and has virtually infinite shelf life prior to final cure.

These and other objects, as well as the scope, nature, and utility of the invention will become more clearly apparent from the following detailed description. Unless otherwise indicated, all proportions and percentages of materials or compounds are expressed on a weight basis throughout the specification and appended claims.

DESCRIPTION OF INVENTION

In accordance with the present invention, durable press garments or similar articles are made by a novel delayed cure technique from cellulosic fiber-containing material, which process essentially comprises applying formaldehyde vapor to the fabric or material in a first stage, preferably in the absence of any catalyst, under conditions which do not greatly increase the original crease resistance of the material (not enough to interfere with subsequent shaping, pressing, etc., and not enough to provide commercially acceptable creaseproofing), washing water soluble substances from the thus treated material if desired, fabricating articles such as garments from the treated material while it remains easy to crease, pleat or otherwise shape, and only then curing the garments or shaped articles in the presence of an acid or acid-reacting catalyst under conditions which cause the cellulose in the material to become creaseproofed as reflected by its greatly improved crease resistance and wrinkle recovery characteristics.

It is possible to carry out the essential formaldehyde pretreatment and to affix the formaldehyde on the material predominantly in a water insoluble polymeric form, e.g., in a form containing about 8 to 100 formaldehyde units per polymer molecule, by exposing the material at a temperature between about 65° and 150° C., preferably between about 110° and 140° C., to a formaldehyde vapor in the substantial absence of any acidic or acid-yielding catalyst and thereby uniformly depositing between about 0.3 percent and 5 percent add-on on the material. In this manner a form of paraformaldehyde is deposited which is not only water insoluble but also remarkably stable. For instance, it is resistant to washing in alkali-containing water at 140° F.

According to a preferred embodiment of the invention even better results are obtained by pretreating the cellulosic material with a monomeric compound which has an active hydrogen and reacts with formaldehyde, e.g., a compound of the amide type, and then forming an amide-formaldehyde condensate rather than a simple polyformaldehyde on the material when it is exposed to formaldehyde vapor as otherwise described above.

Suitable amide-type formaldehyde binding compounds include various ureas such as urea proper, butyl urea, ethylene urea, cyclic propylene urea, allyl urea, cyclic dihydroxyethylene urea, cyanuramide (melamine), thiourea, as well as formamide acetamide, malonamide, acrylamide, the lower alkyl (e.g., C_1–C_4) or hydroxylakyl carbamates such as ethyl carbamate and hydroxyethyl carbamate, aryl sulfonamides such as benzene-sulfonamide or p-benzene-disulfonamide, the lower (e.g., C_1-C_4) alkyl sulfonamides or bis-sulfonamides such as methane-, ethane-, n-butane- or isobutane-sulfonamide, methylene-bis-methane-sulfonamide, ethylene-bis-methanesulfonamide, 1,3-propane-bis-methanesulfonamide, and so on. Urea and ethylene urea have given the highest wrinkle recovery values in the work done to date and are therefore particularly preferred.

When such a formaldehyde-binding compound is used it is particularly advantageous to impregnate the fabric with an aqueous solution of such compound so as to produce a dry add-on of between about 1 percent and 25 percent by weight on the fabric prior to exposure to the formaldehyde vapor. The amide-pretreated fabric is then exposed to formaldehyde in the absence of any acidic catalyst or compound under conditions which are conducive to substantially complete utilization of the N–H groupings of the amide without producing an increase in wrinkle recovery of a magnitude that would interfere with subsequent garment fabrication. Such a reaction of the amide with the formaldehyde forms a substantially water insoluble amide-formaldehyde condensate in situ in the fabric without any pronounced creaseproofing of the cellulose.

A representative, preferred procedure includes applying the amide compound as an aqueous solution preferably having an alkaline pH of less than about 11, e.g., between about 8 and 9, and containing about 1 to 20 percent, e.g., about 10 to 15 percent, of the amide. After padding with such a solution the resulting fabric is desirably partially dried to a moisture content of between about 7 and 15 percent and in this condition then introduced for about 4 to 20 minutes, e.g., 5 minutes, into fumigation chamber maintained at a temperature of between about 105° and 130° C., and filled with an atmosphere consisting essentially of formaldehyde vapor. Optimum exposure time and temperature depend somewhat on factors such as fabric weight and formaldehyde vapor concentration but are readily determined by routine preliminary tests. Air or any other non-acidic gas which does not react with the formaldehyde or the amide and does not catalyze their reaction with cellulose may be present in the formaldehyde fumigation chamber.

A qualitative indication of substantially complete utilization of the N–H groupings in this treatment can be obtained by observing whether the fabric having the amide-formaldehyde condensate formed thereon suffers any noticeable chlorine damage when properly cured and exposed to chlorine bleach. When utilization of the N-H groupings is substantially incomplete, N-chloro compounds are formed on the fabric by reaction with the bleach and when such a fabric is then washed and ironed visible scorch damage and a noticeable loss in tensile strength take place. Of course, a fabric having an amide-formaldehyde condensate deposited thereon will always be more likely to scorch before the fabric has been properly cured in the presence of an acidic catalyst than after such a cure.

After the formaldehyde has been affixed on the material as described above, it is preferred to wash the material in hot water with any conventional detergent in order to remove water soluble compounds and to dry it at any convenient manner, e.g., 80° C. in an air oven. In the absence of any acid the treated material is quite insensitive to temperature and not likely to cure prematurely.

At this point, the present process provides two different options. According to one option the fabric may be padded or otherwise impregnated with a latent solid acid catalyst such as a 1–4 percent aqueous solution of zinc chloride, ammonium chloride, sodium dihydrogen phosphate, magnesium chloride, or the like and dried under non-curing conditions. A protonic acid such as phosphoric acid is also usable as a catalyst, but only when relatively short storage life is required. After drying, the fabric is then ready for use in garment manufacture (e.g., cutting, sewing and pressing) either immediately or after shipment to a different location or after storage of indefinite duration, if desired. Finally, the resulting garments may be made shape retentive by baking in an oven at a temperature between about 90° and 175° C., preferably between about 100° and 160° C., for a time of between about 1 and 30 minutes, preferably at a temperature between about 150° and 160° C. for from 2 to 10 minutes. The optimum reaction time under any given set of reaction conditions is one which is just long enough to effect the desired degree of creaseproofing without weakening the fabric by overexposure to the reactants at the elevated temperature.

According to the other option, the present invention permits making garments from the cellulosic fabrics after formaldehyde treatment, washing and drying, without previous impregnation with any catalyst, the desired cure of the completed garments being effected in This case by exposing the garments to an acidic vapor such as gaseous sulfur dioxide, hydrogen chloride or formic acid in the baking chamber at a temperature and for a time within the same ranges as described above in connection with the first option. Moisture control, e.g., addition of steam, may be used to speed up the curing reaction when sulfur dioxide is used as catalyst, this feature being more fully described in copending application Ser. No. 706,792, filed Feb. 20, 1968, which is commonly owned with the present application and the pertinent portions of which are hereby incorporated by reference in this specification. This second option has the advantage that by employing a catalyst only subsequent to garment fabrication the flat fabric does not contain any catalyst and consequently has a virtually infinite shelf life with very little risk of a premature cure. In contrast, fabrics treated with a latent catalyst may become prematurely cured at least to a partial extent if stored under adverse conditions, e.g., relatively high temperature and high humidity.

The inclusion of polymeric additives which are adapted to form soft films on the fabric can also be included in the process of the present invention to give a further improvement in crease retention and other fabric properties. Suitable polymeric additives include solid resinous or rubbery butadiene-acrylonitrile copolymers, polymers of alkyl acrylates, polyethylene, deacetylated copolymers of ethylene and vinyl acetate, polyurethanes and so forth, as is otherwise well known in the art. Such polymers may be applied to the fabric in the form of an aqueous latex or dispersion of finely divided polymer particles either prior to the formaldehyde vapor treatment or afterwards. In either case, the aqueous polymer dispersion may be padded onto the fabric either by itself or as part of a bath containing some other agent, e.g., an amide prior to the formaldehyde treatment or a solid salt catalyst after the formaldehyde treatment. The polymers are commonly available in commerce in the form of concentrated aqueous dispersions which can be diluted with additional water and can conveniently be included in padding bath to provide therein a solid polymer content of between about 1 and 5 percent, e.g., 3 to 4 percent.

The present invention is useful for treating various natural and artificial cellulosic fibers, as well as various mixtures of such fibers which each other or with other fibers. Natural cellulosic fibers to which this invention is applicable include, for instance, cotton, linen, and hemp. Useful regenerated or artificial cellulosic fibers include viscose rayon and cuprammonium rayon. Other fibers which may be used in blends with one or more of the above mentioned cellulosic fibers are, for example, cellulose acetate, polyamides, polyesters, polyacrylonitrile, polyolefins, polyvinyl chloride, polyvinylidene chloride, polyvinyl alcohol as well as proteinaceous fibers such as wool. As applied to blends, the cellulose crosslinking achieved by the present process gives particularly good results when such blends contain at least 20 weight percent, and most preferably at least 40 weight percent, of cotton or other cellulosic fiber. However, the process of the present invention gives useful results, e.g., dimensional stability, even in the case of fabrics such as wool or wool-polyester blends which do not contain any cellulosic fibers.

The fabric may be knit, woven, non-woven or otherwise constructed. The fabric may be flat, creased, pleated, hemmed or formed into any desired configuration prior to the curing step. After curing, the resulting crosslinked fabric will maintain the desired configuration substantially for the life of the article, e.g., a durable press, wash-wear garment can thus be produced.

The required formaldehyde vapor may be generated in any convenient manner, such as by heating a suspension of paraformaldehyde in mineral oil to generate formaldehyde gas which is then metered into the treating chamber.

Any suitable means may be employed to contact the fabric with the gaseous formaldehyde. For example, a batch system utilizing a closed vessel or tube containing a supply of gaseous formaldehyde may be used into which the fabric to be treated may be placed for an appropriate time. In the alternative, a dynamic or continuous system can be used such as one wherein a stream of formaldehyde is passed through a closed elongated chamber through which the fabric is also passed at an appropriate rate, either concurrently or countercurrently relative to the gas stream. It is also possible to use a combination of the above, that is, one may pass a stream of formaldehyde-containing gas over stationary fabric.

DESCRIPTION OF SPECIFIC EMBODIMENTS

The present invention will now be further illustrated by detailed examples.

The reactor used in this work was a cylindrical vessel having a capacity of about 71 liters, constructed of one-eighth-inch aluminum (42 cm. inside diameter and 57 cm. high). The walls of this reactor are heated with band heaters equipped with a three-way switch which permits operation at 600, 1,200 or 2,400 watts. The reactor wall temperature is controlled by an adjustable bimetallic thermostat, and the reactor is surrounded by an insulating blanket. The gas content of the reactor is recirculated through an external recycle line by an aluminum pressure blower equipped with a heat slinger and high-temperature, lubricated sealed bearings. An adjustable damper in the recycle line permits some control of the pressure within the reactor, but during normal operation nearly atmospheric reactor pressure is preferred.

Formaldehyde gas is conducted into the reactor through heated lines from a separate vessel where it is generated as needed by heating a suspension of paraformaldehyde in mineral oil. The reactor is further equipped with another line through which other gases such as steam, air and/or sulfur dioxide may be admitted when and as desired. The rate of flow of formaldehyde was controlled by regulating the temperature of the mineral oil between 100° and 140° C.

EXAMPLE 1

Formaldehyde Durable Press

A 12 ×13 inch sample of 80 ×80 cotton printcloth was placed on a small frame and immersed into the aluminum reactor described above for one minute while maintaining a formaldehyde flow rate of 3 liters per minute and while maintaining the reactor wall temperature at 120° C. When removed from the reactor, the fabric was washed in a home laundry machine with one cup of "Vel" alkylbenzene sulfonate household detergent powder, and dried. This formaldehyde treated fabric had a dry crease recovery of 181° (W & F) as against 179° (W & F) for an untreated control sample. It is evident that at this stage of the process the shape holding characteristics of the fabric were not materially altered.

Subsequently, the formaldehyde treated fabric was padded with a solution containing 2 percent zinc chloride and 3.2 percent Rhoplex K-14 acrylic resin at 70 percent wet pick-up. The resultant fabric was then dried at 60° C. for 6 minutes and cured at 160° C. in an air oven for 5 minutes. After this cure, the fabric exhibited excellent shape holding properties as indicated by a dry crease recovery of 303° (W & F) and a wash-wear rating of 4.5. When creased, pleated or cuffed prior to the cure, the resulting creases, pleats and cuffs became permanently set upon curing.

EXAMPLE 2

Formaldehyde-Urea Durable Press

In Step 1, a series of 8 oz. cotton twill fabrics were padded at 70 percent wet pick-up with solutions containing 5 to 25 percent urea at pH 8.5. The fabrics were dried and conditioned at 65% relative humidity followed by a 5-minute exposure, in the apparatus of Example 1, to formaldehyde vapor at 120° C. The fabrics were washed in a home laundry machine containing one cup of "Vel" detergent and dried.

In Step 2, each of the samples from Step 1 was padded from a solution of 2 percent zinc chloride and 3 percent Rhoplex K-87 acrylic resin at 70 percent wet pick-up. The resultant fabrics were dried at 80° C., ironed to set a crease, and cured at 160° C. for 7.5 minutes.

The following properties were recorded:

	STEP 1		STEP 2	
Pretreat Solution, Urea %	Crease Recovery (W+F) Wet	Dry	Crease Recovery (W+F) Wet	Dry
5	247	262	298	316
10	220	212	308	325
15	231	216	296	314
20	205	172	285	300
25	220	183	276	290
Control (Untreated)	175	171	—	—

As is evident from these results, Step 1 alone produces a measurable modification of fabric properties, but not enough to interfere with subsequent garment fabrication and far from enough to produce commercially acceptable durable press properties. However, after Step 2 excellent crease recovery is obtained.

Approximately equivalent performance was noted for all these samples independent of urea concentration in the pretreat solution, with the exception that substantial chlorine damage was observed where a high concentration of urea was employed (and the fabric treated with chlorine bleach prior to being treated with catalyst and cured.) A high proportion of urea in the fabric leads to an incomplete polymerization and incomplete utilization of the N-H groupings, unless the formladehyde exposure is appropriately extended and the resulting adduct fully cured in the presence of an acid acting catalyst such as zinc chloride. The paraformaldehyde present on the fabric after the formaldehyde exposure apparently completes the resinification of the urea in the catalyzed curing step and substantially contributes to the large appreciation in crease recovery brought about by such cure. In any event, a minimum add-on of about 0.3 percent is required if formaldehyde alone is used as the creaseproofing agent, or an add-on of at least 1.0 percent is required if a formaldehyde-amide adduct is formed on the fabric, in order to obtain commercially satisfactory performance.

Instead of drying the fabrics after the urea treatment in Step 1 as described above, it is possible to take the wet, urea impregnated fabrics and expose them directly to the gaseous formaldehyde. The course of the reaction and the results obtained are substantially the same.

Unlike in the case of conventional aminoplast resin treatments, fabrics treated in accordance with this invention retain good crease recovery even after acid scouring.

EXAMPLE 3

Another series of treatments similar to those described in Example 2 was run, except that in this series each fabric sample was pretreated in Step 1 with a 15 percent solution of a urea compound, with or without a film-forming polymeric additive, and was padded in Step 2 with a solution containing zinc nitrate as a latent catalyst and in some cases also a film-forming polymeric additive, as indicated. All samples were cured 5 minutes at 160° C. Representative data are summarized in Table I. A more complete tabulation of our data from this work may be found in Textile Research Journal, 38, 414, Table IX (1968).

The adduct formed between the urea compound and the formaldehyde as a result of the non-catalytic formaldehyde treatment in Step 1 was stable to laundering, and, inasmuch as no strong acid catalyst was used, the fabrics were not weakened by such treatment. After the final cure, marked increases in wrinkle recovery values were observed. Permanent creases or pleats could be effectively pressed into these fabrics at any time after application of the latent catalyst and before the high temperature cure.

As shown in Table I, the treated and cured fabrics show high wash-wear and crease retention ratings along with extended wear life as reflected in the laundering test. The fabrics pretreated with urea were exceptionally white, almost as though an optical brightener had been used. The tear strength of these fabrics was high and flex abrasion resistance good. Their tensile strength retention was in the range between 70 and 75 percent as contrasted to 50–60 percent retention for standard pad-bake resin treatments.

Sample cuffs made in accordance with this procedure retained excellent crease recovery after 10 launderings, indicating a stability for the finish which is not characteristic of urea-formaldehyde treatments done by conventional pad-bake procedures.

Moreover, the fabrics treated in accordance with the present invention may be treated with chlorine bleach without **extensive damage, in contrast to fabrics which have been subjected to conventional urea-formaldehyde treatments. Chlorine retention of fabrics treated and cured in accordance with this invention ranges from none to moderate.**

For optimum results the amide pad bath used in the pretreatment should have a pH value of not less than 6, preferably between about 7 and 10. At more acid pH values tensile losses of up to 30 percent are found after the initial formaldehyde treatment. Under optimum conditions of urea deposition any tensile loss is usually not more than about 10 percent. Polymeric additives or softeners, as already state, contribute to performance and, as illustrated in Example 3, may be added either with the amide in Step 1 or with the catalyst solution prior to the catalyzed cure.

TABLE I–PROPERTIES OF PRINTCLOTH TREATED BY TWO-STAGE PROCEDURE

First treatment, pad bath composition	Second treatment pad bath composition*	Reaction time, min.	Add-on after one wash, percent	Add-on after 10 washes, percent	Wrinkle recovery W+F, deg		Tear strength g	Stoll flex abrasion, cycles	Laundering cycles to major damage	Wash-wear rating	Crease retention rating
					Dry	Wet					
Untreated printcloth					180	130	810	400			
Urea (15%)	Catalyst only	5	12.3	–	293	258	460	190	–	–	–
Urea (15%), Rhoplex K-87 (3%) [1]	Catalyst only	5	19.0	17.5	303	261	580	570	8.7	5	4½
Urea (15%)	Rhoplex K-87 (3%) [1]	5	10.8	10.3	296	264	560	290	7.6	4½	5
Urea (15%), Urethane latex E502 (3½%).	Catalyst only	5	14.3	11.4	318	282	600	400	9.9	**5	5
Urea (15%), Mykon SF (3%) [2]	Catalyst only	5	17.4	14.9	304	264	620	510	7.9	4½	4
Urea (15%), Mykon SF (3%) [2]	Catalyst only	3	12.6	–	306	284	660	300	–	5	5
Ethyleneurea (15%)	Catalyst only	5	12.7	–	282	238	560	170	–	–	–
Ethyleneurea (15%), Rhoplex K-87 (3%).	Catalyst only	5	10.4	7.5	311	291	420	390	8.7	4½	4½
Ethyleneurea (15%), Urethane latex E502 (3½%).	Catalyst only	5	8.6	5.8	327	302	440	430	9.9	**5	5
Ethyleneurea (15%)	Urethane latex E502 (3½%)	5	12.0	10.8	308	283	620	460	8.6	4½	5
Ethyleneurea (15%), Hycar 1562 (3%). [3]	Catalyst only	5	5.3	2.6	300	271	380	590	7.7	**4½	4½
CONVENTIONAL "Permafresh" treatment with polyacrylate softener.	–	5	7.5	–	320	270	430	220	3.4	**5	5

* All second-treatment baths contained 2% zinc nitrate hexahydrate. Fabrics were washed before second treatment.

** Fabrics distinctly yellow.

– Means measurement not made.

[1] Acrylic resin. [2] Polyethylene. [3] Butadiene-acrylonitrile copolymer.

EXAMPLE 5

Example 1 was repeated except that the cotton printcloth was pretreated with urea similarly as in Example 2 before the formaldehyde treatment and was not padded with any catalyst in Step 2. Instead, after ironing to set a crease in the formladehyde-treated fabric, it was cured at 115° C. for 20 minutes while passing 200 cc. (measured at 24° C.)/min. of SO_2 and 3 g./min. of steam through the baking oven.

At the end of this cure the fabric had a wet crease recovery (W+F) of 294° and a dry crease recovery (W+F) of 304°, indicative of excellent creaseproofing properties.

It should be understood that while the foregoing specification describes the general principles and nature as well as preferred embodiments and modifications of the present invention, still other modifications are possible and may be made by those skilled in the art without departing from the scope and spirit of this invention. The invention for which protection is sought is particularly pointed out and claimed in the appended claims.

We claim:

1. A deferred-cure, durable-press process for making cellulosic fiber-containing fabric shape retentive, wrinkle resistant and smooth drying by applying a creaseproofing agent to a cellulosic fiber-containing fabric before fabrication of a garment therefrom and then curing said garment after fabrication thereof, which process comprises the sequential steps of:

a. contacting said cellulosic fiber-containing fabric with formaldehyde vapor at a temperature between about 65° and 150° C. until a creaseproofing amount of polymeric formaldehyde in substantially water insoluble form is affixed on said fabric in the absence of an acidic catalyst and without effecting sufficient cross-linking between formaldehyde and cellulosic fiber to produce an increase in wrinkle recovery high enough to interfere with subsequent shaping of the formaldehyde polymer-containing fabric,

b. fabricating a garment from said formaldehyde polymer-containing fabric by steps including cutting and sewing, and

c. heating the resulting garment in the presence of an acidic catalyst under curing conditions at which formaldehyde reacts with the cellulose and improves its wrinkle resistance.

2. A process according to claim 1 wherein the acidic catalyst employed in step (c) is gaseous sulfur dioxide.

3. A process according to claim 1 wherein the acidic catalyst employed in step (c) is formic acid.

4. A deferred-cure, durable-press process for making cellulosic fiber-containing fabric shape retentive, wrinkle resistant and smooth drying by applying a creaseproofing agent to a cellulosic fiber-containing fabric before fabrication of a garment therefrom and then curing said garment after fabrication thereof, which process comprises:

a. contacting said cellulosic fiber-containing fabric and formaldehyde vapor at a temperature between about 65° and 150° C. until a crease-proofing amount of polymeric formaldehyde in substantially water insoluble form is affixed on said fabric in the absence of an acidic catalyst and without effecting sufficient cross-linking between formaldehyde and cellulosic fiber to produce an increase in wrinkle recovery high enough to interfere with subsequent shaping of the formaldehyde polymer-containing fabric,

b. thereafter washing the fabric to remove water soluble deposits therefrom,

c. fabricating a garment from said washed formaldehyde polymer-containing fabric by steps including cutting and sewing, and

d. heating the resulting garment in the presence of an acidic catalyst under curing conditions at which formaldehyde reacts with the cellulose and improves its wrinkle resistance.

5. A process according to claim 4 wherein said formaldehyde polymer-containing fabric is impregnated with an aqueous solution of an acidic curing catalyst and dried subsequent to its being washed and prior to fabrication of garments therefrom.

6. A process according to claim 4 wherein the formaldehyde polymer-containing fabric is impregnated with an aqueous solution of a catalyst which is a solid selected from the group consisting of zinc chloride, ammonium chloride, sodium dihydrogen phosphate, magnesium chloride and phosphoric acid, and dried prior to fabrication of garments therefrom.

7. A deferred-cure process according to claim 4 wherein the cellulosic fiber-containing fabric is impregnated with an aqueous solution of a water soluble amide at an alkaline pH of less than about 11 and dried to produce a dry amide add-on in an amount of between about 0.5 to 25 percent by weight of dry fabric prior to exposure to formaldehyde vapor.

8. A process according to claim 7 wherein the amide is urea and is applied from an aqueous solution at a pH of between about 8 and 9.

9. A process according to claim 4 wherein the garment fabricated from the formaldehyde polymer-containing fabric is cured in a curing zone at a temperature between about 90°

and 160° C. in an atmosphere containing a vaporous acidic curing catalyst until the cellulose becomes creaseproofed.

10. A process according to claim **9** wherein the atmosphere in the curing zone contains about 0.1 to about 50 volume per cent sulfur dioxide and an effective amount of moisture.

11. A process for the sensitizing of cellulosic fiber-containing fabrics to make them suitable for use in the manufacture of garments which are to be made shape retentive by a hot cure subsequent to their manufacture, which process comprises contacting the fabric with formaldehyde vapor at a temperature between about 105° and 140° C. in the absence of catalyst until at least about 0.3 and up to about 5 percent by weight of polymeric formaldehyde in substantially water insoluble form is affixed to the cellulosic fabric based on dry fabric weight without effecting sufficient cross-linking between formaldehyde and cellulosic fiber to produce an increase in wrinkle recovery high enough to interfere with subsequent shaping of the formaldehyde polymer-containing fabric, thereafter washing the fabric to remove water soluble deposits therefrom, drying, impregnating with an aqueous solution of a latent curing catalyst and drying again under non-curing conditions.

12. A process according to claim **11** wherein the cellulosic fiber-containing fabric is impregnated with an aqueous solution of a water soluble amide at an alkaline pH of less than about 11 and dried to produce a dry amide add-on in an amount of between about 0.5 to 25 percent by weight of dry material prior to exposure to formaldehyde vapor.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,653,805 Dated April 4, 1972

Inventor(s) Jose P. Gamarra, Ronald Swidler and Katherine W. Wilson

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

On the page containing the Abstract, Item No. [72] change "Swindler" to -- Swidler -- .

Signed and sealed this 29th day of August 1972.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents