

[54] **PRESS-FREE GARMENT PRODUCTION**

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[58] Field of Search **8/116.2, 116.3; 38/144; 2/243; 260/29.4, 29.1**

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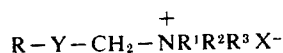
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[57] **ABSTRACT**

Press-free garments are produced by treating a fabric with a mixture of (A) an aldehyde donor compound comprising an amine-aldehyde compound and (B) a synergistic catalyst comprising (1) an acid salt of a primary aliphatic amine and (2) a quaternary ammonium salt of the formula:



wherein

wherein R is an aliphatic hydrocarbon chain of 12 to 18 carbon atoms, Y is —CONH— or —O—, R¹, R² and R³ are alkyl groups or with the nitrogen atom form a substituted or unsubstituted heterocyclic ring and X⁻ is a halogen atom or mineral acid radical; or an alkylaryl N-alkyl sulfonamide. The treated fabric is then partially dried, cut, sewn, pressed and/or creased to form the garment and then cured at an elevated temperature; or dried, cured and then cut and sewn into the garment.

18 Claims, No Drawings

PRESS-FREE GARMENT PRODUCTION

CROSS-REFERENCES TO RELATED APPLICATIONS

This application is a continuation-in-part of my copending application Ser. No. 542,742, filed Apr. 15, 1966, now U.S. Pat. No. 3,458,869.

BACKGROUND OF THE INVENTION

FIELD OF THE INVENTION

This invention relates to textile finishing procedures in general and more particularly to finishing compositions and improved processes for their use in the production of press-free, crease-retaining garments and the products produced thereby.

In recent years, various methods have been developed in the art for treating fabrics so as to impart durable crease characteristics to the fabrics. In the early stages of these developments, processes were discovered which resulted in the so-called "wash and wear" fabrics, the products thereof requiring little or no ironing to reset the crease and remove wrinkles after each washing. The standard process for transforming fabrics into "wash and wear" garments is often called the "pre-cure" technique and generally comprises initial treatment of the fabric with a thermosetting resin, drying, and then curing the impregnated fabric by heating to effect cross linking of the resin and fibers. Thereafter, the fabric is washed, cut, sewn and pressed to result in the finished garment.

Further developments in the garment industry soon gave rise to improvements or variation over the above-defined general process resulting in the so-called "permanent-press" fabrics. In this later development, the fabrics were subjected to an initial treatment with a thermosetting resin and then partially dried. However, in the "permanent press" process, the impregnated fabric, after partial drying, is cut, sewn and pressed to form the completed garment, without washing and prior to curing. Thereafter the finished garment is cured by the use of heat to effect setting or cross-linking of the resin with fibers of the finished garment. This process results in the production of garments which retain their press and remain wrinkle-free even after repeated washings and is called the "post-cure" technique.

While both the "wash and wear" and "permanent-press" techniques have met with general acceptance in the garment industry, and more importantly with consumers, a number of problems have arisen with their use which have precluded universal acceptance and satisfaction of the garments produced by the process. Foremost among these problems or difficulties is that the procedure has not been found suitable for producing press-free garments composed entirely of cotton, particularly so-called light cottons. While some success has been attained by employing fabrics consisting of heavy cottons, i.e., 11 ounces in weight or more, no process has been developed heretofore which will effect suitable durable crease resistance in fabrics consisting of light cottons.

This problem appears to arise because the thermosetting resins employed heretofore for impregnation of the fabrics and the chemicals employed as catalysts for effecting the cross-linking reaction have affected the tensile strength and durability of the fabrics with resultant poor garment wear and discoloration. Hence a great deal of effort has been expended to find a resin/catalyst combination which overcomes these problems of the prior art and is useful in both the "pre-cure" and "post-cure" processes.

As cotton is one of the most desirable fabrics for garment formation, attempts have been made to circumvent the problems by employing cotton, which is composed of cellulosic fibers, fortified with synthetic fibers such as polyesters and polyamides. However, it has been found that incorporation of synthetic fibers into cotton does not effectively prevent or eliminate the deteriorative effect of the finish to the cellulosic fibers, but merely serves to mask the deterioration.

A further problem encountered by prior processes is that the extended curing cycles heretofore employed (16 minutes or more) have resulted in shade changes of many of the dyed fabrics. This difficulty has been particularly manifest when working with fabrics colored by dispersed dyes as the dye-stuff tends to migrate from the cloth to the wall and other parts of the equipment thus resulting in difficult cleaning and maintenance problems as well as creating shading changes and non-uniform shading. Moreover, these prior processes have not been suitable for producing white cottons by reason of the fact that white garments tend to suffer discoloration and yellowing during the curing operation and subsequent wash and wear period. Another difficulty connected with the problem is that the expensive equipment required to effect these long curing or setting periods has mitigated against cost reductions in carrying out the overall process. Another disadvantage resulting from the extended curing cycles of the art is that wash resistance and light fastness properties of the fabric are adversely affected by long, high temperature curing.

Still another disadvantage of processes employed heretofore is that full resin polymerization or cross-linking is not uniformly achieved as the extent and ratio of the inter- and intra-molecular cross-linking may not be preferentially controlled at will.

In my copending above-identified application, many of these problems have been overcome by the provision of a "permanent press" process wherein the fabric is initially treated with an aqueous solution of an aldehyde such as formaldehyde, glutaraldehyde, glyoxal, dialdehyde, para-formaldehyde or aldehyde donor compounds in combination with a synergistic catalyst comprising a salt of a primary amine and a quaternary ammonium ether or amide. In this process however, it was generally believed necessary to incorporate a buffer additive to minimize degradation of the cellulose due to the acid catalyst present. Hence, this process, in many instances, required the use of two different compounds in order to carry out the process, which requirement imposed an economic burden which the present invention obviates.

SUMMARY OF THE INVENTION

It is accordingly one object of the present invention to provide processes and finishing compositions for producing press-free, crease-retaining garments and the products thereof as well as pre-cured garments which obviate the disadvantages heretofore incurred by the art.

A further object of the present invention is to provide an improved process for the production of wash and wear and press-free, crease-retaining garments which retain their durability and tensile strength and are not subject to color variations during production or when subjected to repeated washings during the wear period.

A still further object of the present invention is to provide improved processes for producing garments of full or part cellulosic fiber content by the treatment thereof with a class of compounds which release aldehydes, or react as such, wherein brief curing cycles are employed to result in fabrics having improved tensile, tear and abrasion strength and durability.

An even further object of the present invention resides in the provision of finishing compositions and processes for the production of wash and wear and press-free, crease-retaining garments by the treatment thereof with finishing solutions of aldehyde-containing amine-aldehyde condensates which are capable of forming resinous materials, and a synergistic catalyst, wherein substantially complete resin polymerization and cross-linking is achieved with excellent control of the system.

Other objects and advantages of the present invention will become apparent as the detailed description thereof proceeds.

In accordance with the present invention, and in satisfaction of the above objects and advantages, there are provided improved finishing compositions for the production of wash and wear and press-free, crease-retaining garments which com-

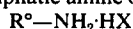
prise a precondensate solution containing an aldehyde donor compound comprising an amine-aldehyde reactant and a synergistic catalyst, the catalyst comprising an acid salt of a primary aliphatic amine and a quaternary salt of an ether or amide or a sulfonamide compound. Also provided by the present invention are the garment products produced and processes for use of the compositions in producing the garments.

DESCRIPTION OF PREFERRED EMBODIMENTS

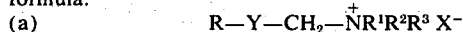
When proceeding according to the concepts of the present invention employing the novel compositions it has been found that treatment of the fabrics with the solution of the aldehyde condensate compound and the synergistic catalyst results in the production of garments having a soft, lustrous hand, outstanding strength and durability properties, remain wrinkle-free and are not subject to discoloration either during the curing step or during the wash and wear period.

The finishing compositions and processes of the invention are considered applicable to the several types of fabrics now employed in the textile industry for forming wash and wear or permanent press garments including 100 percent cellulosics (cotton), 100 percent synthetic fibers as well as blends or mixtures of these fibers, such as cotton blended with nylon or polyesters in ratios of about 40 to 60 percent. Also the invention may be used with wool either as mixed yarns or intimate blends. However, the invention is considered particularly applicable to fabrics composed solely of cellulosic fibers, sometimes called "all-cottons," and this forms a preferred aspect of the present invention. Even more preferred fabrics for use in the present invention are the light-cottons, or the all-cotton fabrics of less than about 11 ounces per square yard in weight. Such light cotton fabrics are eminently suitable for forming garments such as shirts, blouses and the like.

In carrying out the process of the invention, the selected fabrics are initially treated or impregnated with the finishing solution containing (A) an amine-aldehyde condensate donor compound or a mixture of aldehydes as hereinafter defined and (B) a synergistic catalyst mixture constituted of (1) an acid salt of a primary aliphatic amine of the following formula:

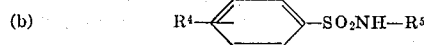


wherein R^o is an alkyl group of one to about six carbon atoms or an aryl-alkyl group of up to about eight carbon atoms and HX is a strong acid; and (2) a member selected from the group consisting of (a) a quaternary ammonium salt of the following formula:



wherein R is an aliphatic hydrocarbon group having about 12 to about 18 carbon atoms and which can be normal or branched, and wherein the carbon chain may contain ethylenic unsaturation or ether bridges; Y is $-CONH-$ or $-O-$; R^1 , R^2 and R^3 are alkyl or aryl groups thus forming a tertiary amine cation, such as dialkylaniline, trialkylamine, etc., or with the nitrogen atom forms a quaternary amine such as pyridinium, quinolinium, picolinium or homologs of these compounds, such as the alkyl-pyridiniums, alkyl quinoliniums, alkyl picoliniums, etc., and X^- is an anion such as a halogen or mineral acid radical, (b) an alkaryl N-alkyl sulfonamide and mixtures thereof.

The alkaryl N-alkyl sulfonamides which may be employed as one of the catalytic components in place of the quaternary ammonium amide or ether (2) above, may be generally described by the following formula:



wherein R^4 is an aliphatic radical, preferably alkyl, having about eight to 20 carbon atoms and R^5 is an alkyl group having about one to 10 carbon atoms. The R^4 group may be in the

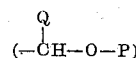
meta-, ortho- or para-position on the phenyl ring but is preferably para to the sulfonamide group. In addition, the R^5 group may be straight or branch chained, e.g., isopropyl, and may further contain one or more non-carbon substituents, such as hydroxyl or alkoxy which will not interfere in the reaction or cause side reactions with the other catalytic components present or the amine-aldehyde reactant employed.

These sulfonamides may be conveniently prepared by reaction of the corresponding alkaryl sulfonic acid or acid halide with an R^5 -substituted amine in the presence of an acid medium with water removal by known methods. A particularly preferred compound of this class is N-isopropyl p-dodecylbenzenesulfonamide.

Use of the sulfonamide reactants in the compositions and processes of the invention represents a particularly preferred aspect as they contribute to the softness and comfort factor of the fabric or garment and thus assist in obviating this disadvantage of similar treated fabrics known to the prior art.

This synergistic catalyst mixture may also contain a third (3) catalyst constituent if desired, namely a salt, e.g., a halide, oxyhalide, nitrate, sulfate, etc., of a metal of Group II, III or IV of the Periodic Table, or an organic acid, which constituent functions to impart a pH below 7 to the treating solution. Any such metallic salt which produces a solution having a pH below 7 may be used. Examples of such salts are the alkaline earth metals, e.g., calcium, barium, magnesium, etc., and particularly the chloride, nitrate, bromide, chlorate and iodide salts thereof; aluminum chloride or sulfate; boron fluoride, soluble zinc salts including zinc silico fluoride; and zirconium oxychloride. Also, there may be employed instead of, or in conjunction with the metal salt, an organic acid such as citric acid, monochloro acetic acid, formic acid and the like with similar results.

The aldehyde-type reactants employed in the compositions and processes of this invention may be generally designated as cellulosic reactant or amine-aldehyde type compounds and/or condensates, the term "amine-aldehyde" meaning that the compounds contain at least one amino group which may be bound to carbonyl and also may contain one or more methylol-type groups



which will react typically to free formaldehyde for reaction with the cellulose hydroxyls under the process conditions. In the above formula for the methylol radical, Q is hydrogen, nitrogen, sulfur or carbon. Obviously when Q is nitrogen, sulfur or carbon, it is to be understood that the grouping is attached to a further portion of the molecule. Also in the above formula P is hydrogen or alkyl of one to about 10 carbon atoms. Exemplary compounds of this class include melamine formaldehyde resin, triazine urea formaldehyde resin (sold commercially as Aerotex 23), dimethylol-ethylene-urea, dimethylol dihydroxyethylene-urea, alkyl-methylol-hydroxyethylene urea, dimethylolformamide, dimethylol methyl carbamate, dimethylol methoxyethyl carbamate, dimethylol diamino anthraquinone, dimethylol ethyl carbamate, dimethylol-n-propyl carbamate, dimethylol acetamide, bis methoxy methylethylene urea, dimethylol hydroxyalkyl carbamate, dimethylol diamino chlorobenzene sulfonic acids, dimethylol N-hydroxyalkyl triazone, dimethylol propylene urea, bis-chloromethylethylene urea, N,N'-methylene bis acrylamide, dimethylol urea paste, dimethoxy methyl uron, dimethylol 5-hydroxy propylene urea, 1-amino-3-chloropropanol, dimethylol 4-methoxydimethyl propylene urea, methoxymethylol hydroxyethyl triazone and the like. Mixtures of these materials may also be employed.

These aldehyde-type reactants used in combination with the catalyst components may be applied from aqueous solutions or from non-aqueous media as the particular media employed is more in the nature of a carrier for the reactant. When a non-aqueous solution is employed, it is preferred to use organic

solvents as the carrier media. Suitable organic solvents which may be employed are alcohols of the aliphatic series containing about one to 10 carbon atoms such as methanol, ethanol, n-propanol, isopropanol, n-butanol, etc.; ketones such as acetone, methyl ethyl ketone, etc.; alkylene glycols such as ethylene glycol; ethers such as petroleum ether, dioxane and tetrahydrofuran; aromatic hydrocarbons such as benzene, toluene, xylene, etc.; aliphatic hydrocarbons such as n-pentane, n-hexane, etc., halogenated aromatic hydrocarbons such as chlorobenzene, ortho-dichlorobenzene, etc., as well as various other solvents such as tetramethylene sulfone, dimethylsulfoxide, butyl oxytol and Stoddards Solvent (Varsol). Also there may be used halogenated aliphatic hydrocarbons such as trichloroethylene, chloroform, carbon tetrachloride and the like. Mixtures of these solvents or with water may also be employed.

The finishing solution may be applied either as a solution or as a mixture such as an emulsion or suspension. Hence, in using the term "finishing solution" herein, it is not intended that the mixture be a true solution as other types of "solutions" may also be employed. Generally when water or a solvent in which all the reactants are soluble are used, the mixture will be a true solution. However, if a water-immiscible solvent is employed or solvent in which all the reactants are not soluble is employed, it is preferred to add an emulsifier in order to provide a homogeneous mixture. This is often necessary since the aldehyde-type reactants are usually available commercially only as aqueous solutions and when a water-immiscible solvent is employed as the carrier, it is preferable to add an emulsifier to the mixture. In this procedure, it has been found according to the invention that when only about 20 percent of water is present in the total solution, up to about 4 percent of an emulsifier when added and the mixture emulsified, will provide emulsions which are so homogeneous that they appear as true solutions to the human eye. Hence use of such emulsified solutions form a significant part of the invention.

In use of the instant compositions true solutions are more desirable than emulsions because more uniform results can be obtained. However, non-aqueous solutions or emulsions are also preferable because of the economy in removal of the solvent on drying.

The amine-aldehyde component is employed in the amount of about 2 to 40 percent by weight of the total solution and more preferably in the amount of about 6 to 35 percent by weight. Aqueous or organic solvent solutions of these materials on a solids basis of about 40 - 60 percent are generally available on a commercial basis.

According to this invention, it has been found that utilization of the amine-aldehyde reactant in the compositions and processes of this invention obviate the need for a reagent to minimize fiber degradation because of the acid catalyst present while still providing excellent tensile strength and crease recovery for the treated products. Moreover, the compositions are applicable to a broad range of fabrics as mentioned above, and further provide excellent hand and wear resistance.

The acid salts of the primary aliphatic amine identified generally hereinabove as component (1) of synergistic catalyst mixture (B), used in conjunction with the amine-aldehydes include the acid salts of compounds of the class defined above such as for example, monoethanolamine; butanolamine; ethylamine; 2-amino-1-butanol; 2-amino-2-methyl-1-propanol; 2-amino-2-methyl-1,3-propanediol; 2-amino-2-ethyl-1,3-propanediol, tris (hydroxy-methyl)-amino methane, etc. The strong acids which may be employed to form the acid salts of the above amines include the mineral acids such as hydrochloric acid, sulfuric acid, nitric acid, phosphoric acid and organic acids such as citric acid, methanesulfonic acid, p-toluenesulfonic acid and the like. The preferred amine salt for use in forming the aqueous solution of the invention is 2-amino-2-methyl-1-propanol hydrochloride, sold commercially under the name Catalyst AC.

The quaternary ammonium component (a) of the class defined above include such compounds as stearamide methyl pyridinium chloride, octadecyl oxymethyl pyridinium chloride, oleyloxymethyl pyridinium chloride, stearamide methyl picolinium chloride, stearamide methyl quinolinium chloride, etc. The preferred quaternary ammonium compound for use in the solution is stearamide methyl pyridinium chloride, sold commercially under the trade name of "Zelan AP," it being preferred chiefly in view of the fact that it is readily available and relatively inexpensive. The compounds described by formula 2(b) are fully described hereinabove.

As mentioned above, the treating solution prepared employing the above-identified materials should contain from about 2 percent to about 35 percent by weight of the amine-aldehyde reactant and not more than about 10 percent by weight of the synergistic mixture of catalysts. The catalyst components in the mixture are preferably present in approximately equal amounts. Generally when operating with or treating cotton fabrics, a smaller amount of the amine-aldehyde component is used than with the other fabrics or fabric blends.

When using a solution containing only catalyst components (1) and (2) above, the amount of the synergistic catalyst mixture should not exceed about 7 or 8 percent by weight based on the total weight of the treating solution. Preferably the solution should contain from about 0.5 percent to about 2 percent of each of the catalyst components (1) and (2) and if component (3) is also used, as preferred, it is employed in the amount of about 1.5 percent by weight to about 2.5 percent by weight of the aqueous solution.

The treating solution may also contain from about 0.5 percent to about 4 percent of glycerin, glucose or sorbitol which serves to protect the fabric, particularly cellulosic fabrics, in the treating solution during the early stages of the processing while the amine-aldehyde is chiefly in the liquid phase. The solution may also contain, if desired, suitable textile softeners as well as suitable deodorants, emulsifiers and the like. Also emulsifying agents such as those based on polyether alcohols may be used to obtain an "optical" solution or an emulsion appearing as a true solution to the human eye, in the presence of water in an amount of up to about 1 - 20 percent when using an immiscible solvent as the carrier as discussed hereinabove.

The above described solution is formed initially to serve as a treating bath for padding or impregnating the fabrics which are ultimately to be formed into garments. In a preferred embodiment, the bath employed for applying the treating solution is prepared by mixing a portion of the neutral solvent carrier with the amine-aldehyde compound and the other constituents are added in solution form to the mixture. The quaternary ammonium salt component is then preferably prepared by dissolving it in water or other suitable solvent at temperatures necessary for forming a solution and adding to the solution. The remaining constituents, if not soluble in water, or carrier used, are dissolved in suitable solvents and added thusly. The temperature of the resultant bath will depend on the particular constituents employed and should be such that all constituents remain in solution. Any temperature below the boiling point of the carrier can be used. As a practical matter, however, temperatures near room temperature or a few degrees above room temperature for example, from 70° F. to about 110° F., should be employed.

In alternative procedures for treating the fabrics with the treating solution, the latter may be sprayed, printed, padded or otherwise applied to impregnate the textile with the treating solution. By either treating method, however, the wet pick-up should be from about 65 percent to about 85 percent by weight based on the weight of the dry textile after extraction of the excess solution.

Before the impregnation of any of the fabrics with the aforementioned solution, it is preferable to thoroughly wash the fabrics in a suitable aqueous soap solution, including a suitable detergent to remove any starch, gelatine, glucose, dextrine, coloring matter or other impurities which may be present, and

subsequently rinsing the washed fabrics in clear rinse water. This procedure is deemed necessary to avoid any undue stiffening of the material. The treated fabrics may then be subjected to either a "pre-cure" or "post-cure" procedure for production of the completed garments as described hereinafter.

Following application of the treating solution to the fabric at the desired temperature and extraction of the excess solution so that the wet pick-up is from about 65 percent to about 85 percent by weight based on the dry weight of the textile, the fabric is dried at a temperature below the temperature required to cure, and in preparation of the permanent press products, well below the temperature required to set or polymerize the resins on the fabric. That is, the polymerization of the resin forming ingredients of the solution, with which the fabric is impregnated, is deliberately avoided in the permanent press method of the invention during the stages of padding, stretching, finishing and drying. A preferred temperature range for drying the impregnated fabric has been found to be about 200° F. to about 260° F. for a period of from about 0.5 to about 3 minutes.

One embodiment for impregnating and drying the fabrics comprises winding the fabric onto rolls and then successively and continuously impregnating with the above-described solution by passing the fabric through a conventional dipping or padding machine, stretching and sizing on a standard tenter frame and simultaneously drying as the fabric passes along the frame. Thereafter, the fabric is rolled onto a suitable drum for transportation to a garment makeup room where the treated fabric is cut, sewn and finished to provide the completed garment for subjection to the final curing step to effect polymerization of the resins in the garment in the permanent press procedure. If desired the fabric may be double-dipped in the above described carrier solution to insure approximately 100 percent pick-up of the solution by the fabric. One variation of the double-dipping involves a drying or drying and baking step followed by reimpregnation of the cloth. Extraction of excess solution may be effected by passing the impregnated fabric through rollers operating under forced pressure so as to result in a wet pickup of about 65 percent to about 85 percent based on the dry weight of the fabric.

In the padding or impregnating step including squeezing of the fabric, approximately 65 to 85 percent, preferably 70 percent, by weight of the solution is retained in the fabric as it leaves the padding machine for passage to the tenter frame for stretching and setting to size such as width. Whenever it is desired to impart a mechanical finish to the fabric, a conventional flat nip calendar can be employed in conjunction with the tenter frame, the rolls of the calendar being heated at the drying temperature to effect the mechanical finish. The drying atmosphere of approximately 200° F. is maintained as the fabric is passed only the tenter frame.

The final roller upon which the fabric is rolled after passing through the tenter frame and flat nip calendar, if used, may or may not be heated depending upon the degree of crease resistance desired in the fabric and this is dependent upon the type of fabric being processed. It has been found that heating of this final or take-off roller usually serves to effect an increase in the crease resistance of the treated fabrics. It is preferred also to so control the drying of the solution and impregnated fabrics so as to retain approximately about 0 percent to about 6 percent of the solution or moisture content over and above the natural moisture of the fabrics. After completion of the impregnation and drying steps, the entire drum or roller with the processed or impregnated fabric, containing the residual moisture content mentioned, is then transferred to a garment preparation location for the usual steps of garment manufacturing, the fabric, as worked upon by the garment makers, containing the unpolymerized or unset resins in the amounts set forth hereinabove when operating by the permanent press procedure.

After the garments have been completed by the steps of cutting, sewing and finishing, which may include pleating or

pressing by a hand or mechanical step if desired, the completed garments are subjected to an elevated temperature to effect complete polymerization and setting of the resin in the garments to a water-insoluble state according to the permanent press or "post-cure" procedure. The curing step employed in setting or polymerizing the impregnated resins according to this invention is preferably conducted at a temperature of about 240° F. to about 350° F. for from about 1 to 10 minutes, preferably about 2 to 5 minutes. For pure cottons the preferred curing temperature is about 300° F. to about 310° F. and for other textiles such as rayons and viscose textiles, the preferred curing conditions are at temperatures of about 320° F. The time required for maintaining these curing conditions to effect complete polymerization is the relatively short period of about 2 to 5 minutes, substantially below the time required in processes employed heretofore. This particularly advantageous short time curing cycle of 2 to 5 minutes represents a significant feature of the process because the shade changes of many dyed fabrics are not adversely affected thereby. Also, the short periods used avoid such problems as non-uniform shading as well as shade changes in the dyed garments. Moreover, when long curing cycles at high temperatures are employed, the web fibers, sensitive to heat, are subject to heat stiffening. Hence, this is also avoided.

The curing operation may be carried out in a number of ways including the use of the conventional curing oven. However, a significant feature of the present process, employing the impregnant solution disclosed herein, resides in the fact that the curing operation may be carried out merely by the application of a heated press. This includes use of the well known "hot-head" press, employed generally in dry-cleaning establishments to effect the pressing of garments. It is to be understood, of course, that the press is to be operated under the time and temperature conditions set out hereinabove.

It is to be understood, however, that the curing process of this invention, employing the aqueous solution described hereinabove, may also be carried out utilizing other heating devices including ovens known as "gas-fired" types. If such ovens are employed, it is necessary that suitable vents be provided therein for permitting the escape of air as well as the escape of vapors emanating from the heated polymerizable resins. Also, the oven should preferably include a plurality of interiorly mounted thermocouples arranged at various locations within the oven for an accurate indication of temperature in the oven as the temperature employed in curing the garments represents a critical feature of the process.

If using the compositions of this invention for producing wash and wear garments, the so-called "pre-cure" technique is employed. In this embodiment, the fabrics, after impregnation with the treating solution and drying as discussed above, are then cured, as described above, and then cut and sewn into the desired garments with pressing and pleating as desired. Preferably, in this procedure, the drying and curing steps are carried out simultaneously, or at least in a single apparatus.

The garments produced as a result of the process of the invention, as hereinabove described, are characterized by having a soft, silky, lustrous hand, outstanding wash resistance, durability and color retention properties and require no ironing after washing, particularly as compared with similar garments produced heretofore. It is theorized that these improvements are due to the unique synergistic catalyst and amine-aldehyde system employed in accordance with the present invention and described hereinabove.

While the explanation for the improvements effected by the synergistic catalyst system is not fully known, the following explanation, believed to be correct, is given to facilitate a better understanding of the invention. It will be appreciated, however, that this invention is not to be limited to this explanation.

The synergistic catalyst system of this invention is believed to provide the most favorable conditions for the reaction between the amine-aldehyde reactant and the fibers with substantially less deterioration of the physical properties of the fibers than has been known heretofore. The components of

the catalyst mixture are believed to cooperate to promote the reaction with optimum yield and with minimum adverse effect on the fiber.

The reaction between the amine-aldehyde and the fiber is believed to take place for the most part during the curing step, as at the curing temperature, reactant aldehyde radical is present for split seconds. At this point, component (1) of the catalyst system provides the acidic pH simultaneously with the release of the reactant radical at the time when the reaction takes place at the curing temperature. The acid salt of the primary aliphatic amine [component (1)] starts to decompose and provides some hydrogen ions at temperatures lower than 200° F.; the major portion, however, does not decompose to form amine vapors and hydrogen ions until temperatures above 240° F., for example, 240° F. to about 350° F., are reacted at which time the major reaction occurs.

In the process reaction, it is believed that the amine-aldehyde in water solution is fugitive and the aldehyde must be kept on the textile where it can react therewith during the curing step in order for the treatment to accomplish the desired results. The acid salt of the primary aliphatic amines is believed to serve as the chemical retainer for the freed aldehyde as it is formed and at the same time provides the necessary acidic conditions when the amine is decomposed during the curing operation to catalyze the reaction between the amine aldehyde and the cellulose.

The quaternary compound or sulfonamide in the synergistic catalyst mixture is believed to promote autopolymerization of the freed aldehyde to less volatile polymerizates which are more reactive with the fibers. These compounds as mentioned above also exercise a desired softening effect on the fabric or garment. The presence of these compounds is particularly important during the early stages of the treatment as their presence in the treating solution or bath insure maximum utilization of the active ingredient. During the curing operation the aliphatic amine appears to exercise the more important catalytic influence in promoting the reaction between the amine-aldehyde and fiber and the two together therefore have a synergistic influence in terms of the overall effect on the textiles, particularly, from the standpoint of imparting to the textiles improved durability and tensile strength properties.

The water soluble metallic salts and/or organic acids employed in the system are considered essential to promote the formation of inter-molecular cross-bonds of the fiber molecules although the intra-molecular cross-bonds are less deteriorative to the fibers than are the inter-molecular types. However, both types of cross-bonds are necessary to provide good durability, garment strength and wash and wear performance. When employing a catalyst mixture containing synergistic components (1) and (2) above and not (3), a high, dry and wet crease angle is produced with less deterioration caused by inter-molecular cross-bonds. In other words, the tensile strength is greater, and while the wash and wear properties are good, they are not as good as is obtained when using a catalyst system containing all three components identified above. As indicated, the use of the latter system results in some loss of tensile strength chiefly because of the formation of more inter-molecular cross-bonds. Component (3) therefore, is believed responsible for more deterioration of the physical properties of the fibers than is component (1) or (2) of the synergistic catalyst mixture. Hence, a proper application of the synergistic catalyst system of this invention permit substantially complete control of the extent and ratio of inter- and intra-molecular cross-links formed as the resin insolubilizes on the fibers. This feature, in turn makes possible the control of the adverse effect of the acidic catalyst on the fibers, particularly with the amine-aldehyde primary ingredients.

The following examples are given to illustrate the process of this invention but it is not to be considered as limited thereto.

In the following examples, 100 percent rayon, 4 sq. yds/lb.; 100 percent cotton sailcloth, 2 ½ sq. yds/lb.; and a blend of polyester cotton containing 65 percent polyester and 35 per-

cent cotton of 4 sq. yds./lb., were treated with the solutions set forth in the examples. The treating solution was at room temperature (70° F.) when the cloth was passed therethrough and after passage through the bite of the padding rollers, the fabric was left with a wet pick-up of about 70 percent based on the fabric weight. The treated cloth was then dried on the rollers at about 200° F.

In the examples, the triazine urea formaldehyde resin (Aerotex 23) employed was used in the form commercially available containing 48 percent solids in an aqueous or isopropanol medium. The dimethylolethylene urea was in the form of a water, alcohol or hydrocarbon solution while some of the methoxylated products were applied from chlorinated hydrocarbon solvents.

Component (1) was Catalyst AC, described above, but the acid salts of any other primary aliphatic amine having six or less carbon atoms such as hydrochloric acid, sulfuric acid, etc., as described above, can be used with similar results. Component (2) referred to as the commercial product "Zelan AP," the compound used being stearamide methyl pyridinium chloride, although other quaternary ammonium salts, as hereinabove disclosed, may be used with equal results. All percentages and values given are by weight based on the weight of the total solution. In addition, in the examples, Mergol SH emulsifier was a sulfated alkyl-aryl polyether alcohol commercially available from Dupont; Synthrapol KB emulsifier was an ethylene oxide condensate commercially available from I.C.I., and Catalyst H—7 is a 30 percent solution of an organic zinc complex neutralized with 2-amino-2-methyl-1-propanol, available from Rohm & Haas.

In the following examples, Examples I to VI illustrate organic solutions or emulsions of the treating materials while Examples VII to XXXVII show use of aqueous solutions in the processes.

EXAMPLE I

Recipe:		
Dimethylol formamide		
solution (50%)	10-25%	
Glycerin	1- 2%	
Aerotex 23	0- 2%	
Zelan AP	2%	
Catalyst AC	2%	
Magnesium Chloride	0-2½%	
Citric Acid	1- 0%	
with methanol	100%	

EXAMPLE II

Recipe:		
N-Methoxy methylethyl		
carbamate	20%	
Dimethylaniline-HCl	2%	
Zelan AP	2%	
Magnesium Chloride · 6H ₂ O	2%	
Monochloroacetic acid	½%	
Isopropanol	73½%	
Total:	100%	

EXAMPLE III

Recipe:		
Bischloromethyl ethylene urea	15%	
Catalyst AC	2%	
Zelan AP	2%	
Zinc Nitrate · 6H ₂ O	1%	
Tetramethylene Sulfone	80%	
Total:	100%	

EXAMPLE IV

Recipe:	
Dimethylol hydroxy ethylene urea	12%
Dimethylol methoxy ethyl carbamate	12%
Zelan AP	2%
Magnesium Chloride	1%
Zinc Nitrate · 6H ₂ O	1%
Stoddard Solvent (Varsol)	64%
Merpol SH (emulsifier)	4%
Synthrapol KB (emulsifier)	4%

Total: 100%

EXAMPLE V

Recipe:	
Dimethylol ethylene urea	22%
Catalyst AC	2%
Zelan AP	2%
Monochlor acetic acid	½%
Butyl oxytol (ethylene glycol monobutyl ether)	73½%

Total: 100%

EXAMPLE VI

Recipe:	
Bismethoxy methyl ethylene urea	20%
Catalyst AC	2%
Zelan AP	2%
Catalyst H-7 (Rohm & Haas)	4%
Merpol SH	2%
Synthrapol KB	2%
Trichloroethylene	68%

Total: 100%

EXAMPLE VII

Recipe:	
Dimethylol methyl carbamate (industrial 50% product)	6-35%

Glycerin	1½%
Aerotex 23	2%
DCY	.25%
Catalyst AC	2%
Calcium Chloride	1%
Tartaric Acid	½- 1%
Zelan AP	2%

with water 100%

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EXAMPLE VIII

Recipe:	
Dimethylol methoxyethyl carbamate (industrial solution 50%)	6-35%
Aerotex 23	2%
Glycerin	1½%
DCY	0-0.5%
Catalyst AC	2%
Zinc Fluoborate	1%
Zelan	2%

with water 100%

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Examples

	IX	X	XI	XII	XIII	XIV	XV
Recipe (percent):							
Dimethylol methyl carbamate ¹	15			6			
Dimethylol ethyl carbamate ¹		25				12	
Dimethylol propyl carbamate ¹			35		20		
Dimethylol acetamide (50% solution)				14			
Dimethylol hydroxyethyl carbamate ¹	10						
Dimethylol methoxy carbamate ¹				16			20
Dimethylol N-ethyl triazone	6	2.5		2		8	
Dimethylaniline · HCl	2	2	2	2	1	2	2
Zelan AP	2	2.5	2	2	2	2	2
Magnesium chloride	2	1.5	2	1	2	1	1.6
Magnesium nitrate		1.5		1		1	
Zinc nitrate							1.5
Citric acid	1				1		
Water	72	65	59	62	74	74	63

50 ¹ Industrial 50% solution.

Examples

	XVI	XVII	XVIII	XIX	XX	XXI	XXII	XXIII	XXIV	XXV	XXVI
Recipe (percent):											
Dimethylol ethylene urea (50% solution) ¹	15				8						
Dimethylol propylene urea (50% solution) ¹			20		10						
Dimethylol dihydroxyethylene urea				25		10		15	8		
Dimethylol urea paste ¹					5						
Dimethylol uron ¹	10					20			15	20	
Dimethylol-5-hydroxy-propylene urea ¹		10									
Dimethylol-4-methoxy dimethyl propylene urea ¹							15				
Dimethylol formamide ¹							12				
Dimethylol ethyl carbamate ¹		12	10					12			20
Aerotex 23		2			2		2				
Dimethylol hydroxyethyl triazone ¹	2		2	2				6		3	
Monoethanol amine · HCl (30%)		2	2		4		2		2		2
Catalyst AC	2			2		2		2		2	2
Zelan AP	2		2	2		2					
Atlas G-263 (cetylamine N-ethyl morpholinium etho-											
sulfate)		2			3					2	
N-isopropyl dodecylbenzene-sulfonamide											2
Ethyl-dimethyl-octadecyl(ethyl hydrogen phosphite)							2	3	2		
Zinc nitrate	1			1			1	1			1
Magnesium chloride	1		2.5			2		1	2	0.5	1
Maleic acid				25							2
Water	69	70	61.5	66.7	68	64	66	60	61	60.5	73.8

¹ Industrial 50% solution.

Each of the above examples were applied to 100 percent rayon, 100 percent cotton and the polyester/cotton mixture described above, dried, made into garments and cured at 300° C. for 2 minutes according to the "post-cure" method described. The resulting garments showed excellent tensile strength, crease recovery, soil resistance and hand.

In the following tables there are presented laboratory test results on a selected number of applications, using for the purpose of demonstration the following different substrates: 100 percent rayon 4 sq. yds/lb., 100 percent cotton sail cloth of 2 ½ sq. yds./lb., and an intimate blend of polyester-cotton containing 65 percent polyester and 35 percent cotton of 4 sq. yds./lb. weight. These experiments were carried out according to the "post-cure" method described above and show the following results using the indicated treating solutions of the previous examples.

TABLE I

100% Rayon 4 sq. yds/lb.					
Example	Tensile		Crease Recovery		No-iron Rating
	Warp	Fill	Warp	Fill	
X	32	30	140	148	4.5
XVI	46	36	128	130	3.5
XIX	39	34	136	140	4.0
XXIII	36	33	138	142	4.5

TABLE II

100% Cotton					
Example	Tensile		Crease Recovery		No-iron Rating
	Warp	Fill	Warp	Fill	
X	50	28	138	140	5.0
XVI	62	35	120	126	4.0
XIX	48	30	130	136	4.5
XXIII	52	29	140	142	4.5

TABLE III

Polyester/Cotton Blend					
Example	Tensile		Crease Recovery		No-iron Rating
	Warp	Fill	Warp	Fill	
X	48	42	140	144	4.5
XVI	52	46	130	136	4.0
XIX	50	44	140	136	4.5
XXIII	52	42	146	150	4.5

In the above Tables, the values for tensile strength were obtained by the Scott Tester ASTM Standard D39-49. The Crease Recovery values were obtained by the Monsanto Method, ASTM Standards D1295-53T American Society for Testing Materials, Philadelphia, Pa. Wash and wear or No-iron Rating was determined by AATCC Tentative Test method, 88-1960. AATCC Technical Manual 1960, Volume 36, page 148. According to this test procedure a value of 5 is perfect and a value of 4 is excellent.

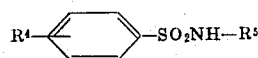
As will be seen in the Tables, the tensile strength and crease recovery are both excellent and generally consistent. Moreover, with one exception, the no-iron ratings are excellent or perfect.

These fabrics, when tested for shrinkage upon repeated washings showed exceptionally good dimensional stability. They had a pleasing soft hand and showed good wear resistance, rendering them particularly useful for shirtings and other apparel of the better grades.

It is to be understood that obvious variations of the treating solutions used herein may be employed in the invention. Hence the invention is not to be considered as limited except by the scope of the appended claims.

What is claimed is:

1. A process for the production of press-free crease-retaining garments which comprises treating a cellulose-containing fabric from which the garments are produced with a finishing solution containing reactive chemicals in a carrier containing (A) about 2 to 40 percent by weight of a reactive amine-aldehyde compound selected from the group consisting of melamine formaldehyde resin, triazine urea formaldehyde resin, dimethylolethylene urea, dimethylol dihydroxyethylene urea, alkyl-methylol-hydroxy ethylene urea, dimethylol formamide, dimethylol methyl carbamate, dimethylol methoxyethyl carbamate, dimethylol diamino anthraquinone, dimethylol ethyl carbamate, dimethylol-n-propyl carbamate, dimethylol diamino-chlorobenzene sulfonic acids, dimethylol N-hydroxyalkyl triazone, dimethylol propylene urea, bischloromethyl ethylene urea, N,N'-methylene bis acrylamide, dimethylol urea paste, dimethoxy methyluron, dimethylol 5-hydroxy-propylene urea, 1-amino-3-chloropropanol, dimethylol 4-methoxydimethyl propylene urea, methoxymethylol hydroxyethyl triazone and mixtures thereof; and (B) up to 10 percent by weight of a synergistic catalyst mixture comprising (1) an acid salt of a primary aliphatic amine selected from the group consisting of the acid salts of monoethanolamine, n-butanolamine, ethylamine, 2-amino-1-butanol, 2-amino-2-methyl-1-propanol, 2-amino-2-methyl-1,3-propanediol, 2-amino-2-ethyl-1,3-propanediol, tris(hydroxymethyl)aminomethane and dimethylaniline; and (2) a member selected from the group consisting of (a) stearamide methyl pyridinium chloride, octadecyloxy methyl pyridinium chloride, oleyloxymethyl pyridinium chloride, stearamide methyl picolinium chloride or stearamide methyl quinolinium chloride and (b) a sulfonamide of the formula



wherein R⁴ is an alkyl group having about eight to 20 carbon atoms and R⁵ is an alkyl group having about one to 10 carbon atoms, or mixtures thereof, drying the treated fabric, cutting and sewing to form the desired garment, pressing and/or creasing the garment and curing the garment at an elevated temperature.

2. A process according to claim 1 wherein the synergistic catalyst mixture also contains a third catalyst component (3) comprising a compound consisting of the halides, oxyhalides, nitrates and sulphates of a metal from Groups II, III or IV of the Periodic Table or an organic acid comprising citric acid, monochloroacetic acid, formic acid or maleic acid.

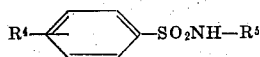
3. A process according to claim 1 wherein the carrier is a water-immiscible organic solvent.

4. A process according to claim 3 wherein the fabric treated is composed of cellulosic fibers or a blend of cellulosic and synthetic fibers.

5. Press-free, crease-retaining garments produced by the process of claim 1.

6. A finishing solution containing reactive chemicals in a carrier for the treatment of cellulose-containing fabrics which comprises (A), an amine-aldehyde compound which will react to free formaldehyde, and selected from the group consisting of melamine formaldehyde resin, triazine urea formaldehyde resin, dimethylolethylene urea, dimethylol dihydroxyethylene-urea, alkyl-methylol-hydroxy ethylene urea, dimethylol-formamide, dimethylol methyl carbamate, dimethylol methoxyethyl carbamate, dimethylol diamino anthraquinone, dimethylol ethyl carbamate, dimethylol-n-propyl carbamate, dimethylol diamino-chlorobenzene sulfonic acids, dimethylol N-hydroxyalkyl triazone, dimethylol

propylene urea, bischloromethyl ethylene urea, N,N'-methylene bis acrylamide, dimethylol urea paste, dimethoxy methyluron, dimethylol 5-hydroxy-propylene urea, 1-amino-3-chloropropanol, dimethylol 4-methoxydimethyl propylene urea, methoxymethylol hydroxyethyl triazone and mixtures thereof; and (B) a synergistic catalyst mixture comprising (1) an acid salt of a primary aliphatic amine selected from the group consisting of the acid salts of monoethanolamine, n-butanolamine, ethylamine, 2-amino-1-butanol, 2-amino-2-methyl-1-propanol, 2-amino-2-methyl-1,3-propanediol, 2-amino-2-ethyl-1,3-propanediol, tris(hydroxymethyl)aminomethane or dimethylaniline; and (2) a member selected from the group consisting of; (a) stearamide methyl pyridinium chloride, octadecyloxymethyl pyridinium chloride, oleyloxymethyl pyridinium chloride, stearamide methyl picolinium chloride or stearamide methyl quinolinium chloride, and (b) a sulfonamide of the formula



wherein R⁴ is an alkyl group having about eight to 20 carbon atoms and R⁵ is an alkyl group having about one to 10 carbon atoms, and mixtures thereof.

7. A finishing solution according to claim 6 wherein the carrier is selected from the group consisting of aqueous media, substantially water-immiscible organic solvents and mixtures thereof.

8. A finishing solution according to claim 7 wherein the solution contains about 2-40 percent by weight of the amine-aldehyde and the total amount of the synergistic catalyst mixture in the composition is up to about 10 percent by weight.

9. A finishing solution according to claim 8 wherein the synergistic catalyst mixture also contains a third catalyst component (3) comprising a compound consisting of the halides, oxyhalides, nitrates and sulphates of a metal from Groups II, III or IV of the Periodic Table or an organic acid selected from the group consisting of citric acid, monochloroacetic acid, formic acid and maleic acid.

10. A finishing composition according to claim 9 which contains 7 to 8 percent by weight of the catalyst mixture and about 6 to 35 percent by weight of the amine-aldehyde with a total solids weight of about 40 to 60 percent.

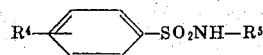
11. A finishing solution according to claim 10 wherein the sulfonamide catalyst component (2-b) is N-isopropyl p-dodecylbenzene sulfonamide or N-isopropyl tridecyl phenyl-sulfonamide.

12. A finishing solution according to claim 10 wherein component (1) of the catalyst system is 2-amino-2-methyl-1-propanol hydrochloride and component (2) is stearamide methyl pyridinium chloride.

13. A finishing solution according to claim 12 wherein the carrier is a water-immiscible organic solvent in combination with an emulsifier.

14. A finishing solution containing reactive chemicals in a carrier for the treatment of cellulose-containing fabrics which comprises (A) about 2 to 40 weight percent of a cellulosic reactant amine-aldehyde compound selected from the group

consisting of melamine formaldehyde resin, triazine urea formaldehyde resin, dimethylol ethylene urea, alkyl-methylol-hydroxyethylene urea, dimethylol dihydroxy ethylene urea, dimethylol formamide, dimethylol methyl carbamate, dimethylol methoxyethyl carbamate, dimethylol diaminoanthraquinone, dimethylol ethyl carbamate, dimethylol n-propyl carbamate, dimethylol acetamide, bis methoxy methylethylene urea, dimethylol hydroxyalkyl carbamate, dimethylol diaminochlorobenzene sulfonic acid, dimethylol N-hydroxyalkyl triazone, dimethylol propylene urea, bischloromethyl ethylene urea, N,N'-methylene bis acrylamide, dimethylol urea paste, dimethoxy methyluron, dimethylol 5-hydroxy propylene urea, 1-amino-3-chloropropanol, dimethylol 4-methoxy-dimethyl propylene urea, methoxymethylol hydroxyethyl triazone and mixtures thereof, and (B) up to about 10 weight percent of a synergistic catalyst mixture comprising, (1) an acid salt of a primary aliphatic amine selected from the group consisting of the acid salts of monoethanolamine, n-butanolamine, ethylamine, 2-amino-1-butanol, 2-amino-2-methyl-1-propanol, 2-amino-2-methyl-1,3-propanediol, 2-amino-2-ethyl-1,3-propanediol, tris(hydroxymethyl)aminomethane or dimethylaniline; and (2) a member selected from the group consisting of; (a) stearamide methyl pyridinium chloride, octadecyloxymethyl pyridinium chloride, oleyloxymethyl pyridinium chloride, stearamide methyl picolinium chloride or stearamide methyl quinolinium chloride, and (b) a sulfonamide of the formula



wherein R⁴ is an alkyl group having about eight to 20 carbon atoms and R⁵ is an alkyl group having about one to 10 carbon atoms, or mixtures thereof; and said carrier is selected from the group consisting of aliphatic alcohols containing about one to 10 carbon atoms, ketones, alkylene glycols, ethers, aromatic hydrocarbons, aliphatic hydrocarbons, halogenated aromatic hydrocarbons, halogenated aliphatic hydrocarbons, tetramethylene sulfone, dimethyl-sulfoxide, butyl oxytol, Stoddards Solvent and mixtures thereof.

15. A finishing solution according to claim 14 wherein the synergistic catalyst mixture also contains a third catalyst component (3) comprising a compound consisting of the halides, oxyhalides, nitrates and sulphates of a metal from Groups II, III or IV of the Periodic Table or an organic acid selected from the group consisting of citric acid, monochloroacetic acid, formic acid and maleic acid.

16. A finishing composition according to claim 15 which contains 7 to 8 percent by weight of the catalyst mixture and about 6 to 35 percent by weight of the amine-aldehyde with a total solids weight of about 40 to 60 percent.

17. A finishing solution according to claim 16 wherein component (1) of the catalyst system is 2-amino-2-methyl-1-propanol hydrochloride and component (2) is stearamide methyl pyridinium chloride.

18. A finishing solution according to claim 16 wherein the sulfonamide catalyst component (2-b) is N-isopropyl p-dodecylbenzene sulfonamide or N-isopropyl tridecyl phenyl-sulfonamide.

* * * * *

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,663,159

Dated May 16, 1972

Inventor(s) Alex F. Gordon

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

In the Abstract, one line beneath the formula, delete
"wherein" first occurrence;

Column 12, Example XV, after Dimethylol ethyl carbamate¹
insert --10--;

Column 12, Examples IX-XV, six lines down, delete
"Dimethylol methoxy carbamate¹" and insert --Dimethylol
methoxy ethyl carbamate¹--.

Signed and sealed this 12th day of February 1974.

(SEAL)
Attest:

EDWARD M. FLETCHER, JR.
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents