United States Patent

Thomas

[54] PREPARING PERMANENT PRESS GARMENTS BY TREATING WITH COMPOSITION THEREFOR

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[21] Appl. No.: 706,605

[52] U.S. Cl. 8/115.6, 8/115.7, 8/116.2, 8/127.6, 117/139.5 R, 117/163, 117/141, 117/161, 117/161 UD, 117/139.4, 8/DIG. 11, 38/144

[51] Int. Cl. D06m 15/28, D06m 15/50

[58] Field of Search 8/116.2, 115.6, 115.7; 117/139.5, DIG. 7

[56] References Cited

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[57] ABSTRACT

A fabric having been impregnated with a composition comprising a neoprene elastomer, a polyisocyanate, polyisothiocyanate, blocked derivatives and mixtures thereof, and a metal oxide are found to have a propensity for subsequent durable dry setting in a preselected configuration. Fabrics which have been prepared and set in this manner exhibit improved crease retention, flat dry stability and resistance to shrinkage even when subjected to home laundering operation.

10 Claims, No Drawings
PREPARING PERMANENT PRESS GARMENTS BY TREATING WITH COMPOSITION THEREFOR

BACKGROUND OF THE INVENTION

This invention relates to a process for improving characteristics of textile fibers, and more particularly, to the preparation of fabrics having a propensity for subsequent durable dry setting.

Garments containing creases which are durable to home laundering operations are known in the art. Garments prepared from cellulosic fiber-containing fabrics having home laundry durable creases set therein have recently found wide acceptance in the industry. Cellulosic garments of the above-mentioned types and the methods for their preparation are set forth in U.S. Pat. No. 2,974,432. Satisfactory processes for the preparation of durable creases in wool fabrics which will stand home laundering operations also have been known, but these processes generally involve some form of wet chemical treatment at the time of setting. For example, one type of wool fabric known to be useful in the preparation of garments having creases durable to home laundering operations is subjected to certain chemical and physical treatments in fabric form, cut and formed into a garment, and thereafter subjected to additional chemical treatments prior to setting. Such a procedure requires that the cutter and garment manufacturer maintain skilled personnel and special equipment for the treatment of these fabrics prior to setting. For these reasons, such methods for preparing permanent press wool fabrics have not been completely accepted in the industry.

Another method for preparing durable creases in natural fiber-containing fabrics has involved the use of blends of thermoplastic fibers and natural fibers such as cotton and wool. By setting the thermoplastic component of the fabric at temperatures near the melting point of the thermoplastic fiber, a crease is produced which has a certain degree of durability to home laundering operations. However, the setting operation generally destroys the desirable hand and surface effects of the fabrics.

Still another method involves a three-blend fabric containing, for example, 50 percent wool, 40 percent rayon, and 10 percent nylon wherein the rayon component is reacted with a typical postcurled resin such as didehydro dimethyl ethylene urea and subsequently pressed and cured in an oven.

SUMMARY OF THE INVENTION

These problems have been overcome by providing textile fabrics having a propensity for subsequent durable setting which have been impregnated with a composition comprising a neoprene elastomer, a compound selected from the class consisting of polyisocyanates; polyisothiocyanates; blocked derivatives and mixtures thereof, and a metal oxide. These impregnated fabrics may then be cut, converted into a garment, maintained in a desired configuration, and thereafter subjected to a temperature sufficient to cure the impregnated fabric. The curing is a dry curing process thereby eliminating any necessity for the cutter and garment manufacturer to further treat the garment before it is pressed in its permanent configuration.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The textile fabrics which are useful in this invention may be prepared from virtually all types of fibers ranging from fabrics containing 100 percent natural fibers such as cotton or wool fabrics composed exclusively of synthetic fibers such as polyesters and polyamides. Blends of these fibers also have been valuable for preparing garments having durable creases.

Further examples of fabrics include those containing natural fibers such as flax, ramie, alpaca, vicuna, mohair, cashmere, guanaco, camel hair, llama, fur, suede and silk. Synthetic fibers include polyamides such as polyhexamethylene adipamide; polyesters such as polyethylene terephthalate; acrylic fibers such as polyacrylonitrile, homopolymers or copolymers of acrylonitrile, such as acrylonitrile/methylacrylate (85:15); and cellulosic derivatives such as cellulose acetate and viscose rayon. The invention is particularly adapted to wool fabrics which heretofore have been particularly difficult to set by post curing in a preslected configuration durable to home laundering.

Examples of fabrics wherein synthetic fibers are blended with natural fibers include wool/nylon (85:15); Acrylan/wool (55:45); Orlon/wool (65:35); Dacron/wool (55:45); wool/rayon (65:35); and wool/rayon/nylon (65:25:10).

Laminated fabrics are also susceptible to improvement by this invention. The laminated fabrics can be composed of two or more layers of fabric bonded together with an adhesive. Such laminated fabrics are well known in the art. The outer layer is generally a woven or knitted fabric comprised of natural fibers, synthetic fibers or blends thereof. Examples of such fibers and fabrics have been listed above. The inner or backing layer of the laminated fabric is preferably a knitted fabric obtained from spun or continuous filament yarns. Particularly useful and popular knitted fabrics include those prepared from such fibers as cotton, nylon, polyester, cellulosic acetate, rayon and viscose rayon. Knitted tricot fabrics such as acetate and nylon tricot have achieved wide popularity.

The neoprenes useful in the composition of this invention are chloroprene based synthetic elastomers. These may be either polymers of chloroprene or copolymers of chloroprene with other polymerizable ethylenically unsaturated compounds, such as acrylonitrile. Copolymerization is generally catalyzed by a potassium persulfate catalyst. The progress of the polymerization is followed by means of specific gravity changes. Depending upon the type of neoprene latex desired, a shortstop may be added after the desired conversion has been obtained, or the polymerization may be carried to completion.

In this manner, a wide variety of neoprenes can be prepared. Examples of solid neoprenes which are available commercially include the "sulfur-modified" types, such as Neoprene Type GN (a sulfur-modified chloroprene polymer stabilized by a thiaurium disulfide) and Neoprene Type GN-A (a sulfur-modified chloroprene polymer stabilized by a thiaurium disulfide and containing a secondary aromatic amine stabilizer), Neoprene Type GRT (a sulfur-modified chloroprene polymer stabilized by a thiaurium disulfide and containing a nondoniscoloring antioxidant), and Neoprene Type W. (a nonsulfur-modified general-purpose type of neoprene). Although both the GN and W Types are made by emulsion polymerization chloroprene, the W type of neoprene has a more uniform molecular structure and does not contain sulfur or other compounds capable of decomposing to yield free sulfur. As a result of these differences, Type W neoprene displays improved storage ability and better processing properties. Magnesium and zinc oxide are required modifying agents in all neoprene Type W formulations.

Neoprene Type Q is a copolymer of chloroprene and acrylonitrile stabilized with a thiaurium disulfide and containing a nondiscoloring antioxidant. These and other neoprenes are described in more detail in the book entitled The Neoprenes, Principles of Compounding and Processing by Neil L. Cattan, published in 1953 by E. I. duPont de Nemours and Co., Wilmington, Del.

Neoprene latices are extremely useful in the preparation of the compositions of this invention, especially in the preparation of aqueous dispersions or emulsions of the compositions. The neoprene latices are emulsions of polymerized chloroprene or copolymers of chloroprene in water, which contain emulsifying agents and stabilizers. The latices are milke-like liquids containing from about 35 to 60 percent total solids. These latices are further characterized in that they do not tend to settle out significantly, even though the neoprene polymer has a specific gravity considerably higher than that of the liquid in which it is suspended. The different types of neoprene latices currently available are obtained by varying certain manufacturing procedures including the emulsification, catalyst and modifier systems. Resulting latices differ
from one another in polymer and colloid properties. A variety of neoprene latices are available from the E. I. duPont de Nemours and Co. and several of these are described in the book entitled Neoprene Latex, Principals of Compounding and Processing, by John C. Carl, 1962. Examples of such neoprene latices include Neoprene Latex 400 (a high modulus and coagulum resisting latex containing about 50 percent solids having a Brookfield viscosity of 15 c.p.s.), Neoprene Latex 750 (a low modulus latex which is highly resistant to crystallization; contains about 50 percent solids and has a Brookfield viscosity of 13 c.p.s.), Neoprene Latex 650 is a concentrated form of Latex 750 containing about 60 percent solids and having a Brookfield viscosity of 4,000 c.p.s. An example of a latex emulsion containing a copolymer of chloroprene and acrylonitrile is Neoprene Latex 450. The copolymer is highly oil resistant and noncrystallizing.

The particular solid neoprene or neoprene latex chosen for the preparation for the compositions of the invention will depend upon the relationship of the properties of the neoprene and those desired of the fabrics treated with the composition. This selection will be apparent to those skilled in the art.

The compositions of this invention also contain a compound selected from the class consisting of polyisocyanates, polyisocyanates, blocked derivatives or mixtures thereof. Polyisocyanates and blocked polyisocyanates are preferred.

The suitable isocyanates that are useful in accordance with this invention include, for example, arylisocyanates, such as 2,4-toluene diisocyanate, 2,6-toluene diisocyanate, 4,4'-diphenylmethane diisocyanate, p-phenylene diisocyanate, 1,5-naphthylene diisocyanate, m-phenylene diisocyanate, diphenyl-4,4'-diisocyanate, 1-isopropylbenzene-3,5-diisocyanate, 1-methyl-phenylene-2,4-diisocyanate, naphthylene-1,4-diisocyanate, diphenyl-4,4'-diisocyanate, 5-nitro-1, 3-phenylene diisocyanate, xylene-1, 4-diisocyanate, xylene-1,3-diisocyanate, 4,4'-diphenylmethane diisocyanate, 4,4'-diphenylmethane diisocyanate, and as well as mixtures thereof and including the equivalent isocyanates. Of these compounds, the aryl isocyanates are preferred because of their solubility and availability.

Additional isocyanates include polyisocyanates, disocyanates and diisocyanates, such as ethylene disocyanate, dimethylene disocyanate, dodecamethylene disocyanate, hexamethylene disocyanate, tetramethylene disocyanate, pentamethylene disocyanate, and diisocyanates; alkylenediisocyanates and diisocyanates such as propylene-1,2-diisocyanate, 2,3-dimethyltetramethylene disocyanate and diisocyanate, butylene-1,2-diisocyanate, butylene-1,3-diisocyanate, and diisocyanates such as ethylene diisocyanate (CH$_2$CNH(NCO)$_2$) and heptylidene diisocyanate (CH$_2$CH$_2$CH(NCN)$_2$); cycloalkylene diisocyanates and diisocyanates such as 1,4-diisocyanatocyclohexane, cyclopentylene-1,3-diisocyanate, and cyclohexylene-1,2-diisocyanate; aromatic polyisocyanates and polysiocyanates such as phenylethylene disocyanate (CH$_2$CH(NCO)(NCO)CH NCO); diisocyanates and diisocyanates containing heteroatoms such as SCNH-CH$_2$CH(NCS), and SC(H)(CH$_2$)$_3$-S—(CH$_2$)$_n$NCS 1,2,3,4-tetraisocyanatobutane, butane-1,2,2-triisocyanate, toluene-2,4,6-trisocyanate, benzene-2,3,4-trisocyanate, benzene-1,3,5-trisocyanate, benzene-1,2,3-trisocyanate, 1-isocyanato-4-isocyanatohexane, and 2-chloro-1,3-diisocyanatopropane.

Thioisocyanates or isothiocyanates may be derived from the corresponding blocked compound in accordance with conventional technology. Blocked isocyanates contain little or no free isocyanate groups as the result of the addition onto these groups by active hydrogen compounds (as determined by the Zerewitinoff method). These addition products are relatively inert at room temperatures but have only limited thermal stability. The blocked isocyanates beyond a certain dissociation temperature, the addition product is activated, or freed and takes part in the curing process.

In the preparation of the adducts, the polyisocyanate and the adduct forming compound are usually dissolved in a suitable inert solvent such as toluene, methyl ethyl ketone, or o-dichlorobenzene. The solutions are stirred together and permitted to stand. The reaction should be caused to take place at a temperature below the decomposition temperature of the desired product and preferably at a temperature not exceeding approximately 100°C. In most instances, the reaction will proceed satisfactorily at room temperature. When the solvent used for the isocyanate compound and blocking agent is not also a solvent for the adduct formed, the adduct formed separates from the solution and is removed therefrom by filtration or evaporation of the solvent. The time required for the adduct to form will vary from a few minutes to several hours depending upon the particular reactants used. The precipitated product will probably contain small amounts of unreacted material which, if necessary, can be removed by recrystallization or extraction procedures known to those skilled in the art.

Preferred adduct-forming compounds produce adducts which may be activated, or unblocked, by heat alone. Typical active hydrogen compounds which provide heat-reversible adducts include the following:

1. Tertiary alcohols, such as tertiary butyl alcohol, tertiary amyl alcohol, dimethyl ethyl carbinal, dimethyl phenyl carbinal, methyl diphenyl carbinal, triphenyl carbinal, 1-nitro tertiary butyl carbinal, and 1-chloro tertiary butyl carbinal;

2. Secondary aromatic amines which contain only one group having a hydrogen reactive with an isocyanate group, such as the diaryl compounds, including diphenyl amine, o-ditolyl amine, m-ditolyl amine, p-ditolyl amine, N-phenyl toluidine, N-phenyl xylidine, phenyl alpha naphthyl amine, phenyl beta naphthyl amine, carbazole, and the nucleus substituted aromatic compounds such as 2,2'-dinitro diphenyl amine and 2,2'-dichloro diphenyl amine;

3. Mercaptans, such as 2-mercaptobenzothiazole, 2-mercapto thiophione, dodecyl mercaptan, ethyl 2-mercapto thiазole, dimethyl 2-mercapto thiazole, beta naphthyl mercaptan, alpha naphthyl mercaptan, phenyl 2-mercapto thiazole, 2-mercapto 5-chloro benzothiazole, methyl mercaptan, ethyl mercaptan, propyl mercaptan, butyl mercaptan, and ethyl dimethyl thicarbinol;

4. Lactams, such as epsilon-caprolactam, delta-valerolactam, gamma-butyrolactam, and beta-propiolactam;

5. Imides, such as carbimide, succinimide, phthalimide, naphthalimide, and glutarimide;

6. Monohydric phenols in which the hydroxyl group is the only group containing hydrogen reactive with the isocyanate group, such as the phenols, cresols, xylanols, trimethyl phenols, ethyl phenols, propyl phenols, chloro phenols, nitro phenols, thymols, mona alpha phenyl ethyl phenol, di alpha phenyl ethyl phenol, tri alpha phenyl ethyl phenol and tertiary butyl phenol;

7. Compounds containing enolizable hydrogen, such as acetoacetic esters, diethyl malonate, ethyl n-buty1 malonate, ethyl benzyl malonate, acetyl acetone, acetonyl acetone and benzimidazole;

The adduct-forming compounds should, of course, possess only one group containing a reactive hydrogen atom. The presence of more than one such group would permit polymerization reactions with the polyisocyanate which are not desired in most instances. Among the more preferable adduct-forming compounds are included diphenyl amine and phenol.

The use of such blocked polyisocyanates is preferred in his invention for a number of reasons which are based on the fact that the blocked isocyanates are not reactive under normal conditions. The selection of such blocked isocyanates permits the use of water as the dispersing medium and avoids the necessity of utilizing organic solvents. Moreover, the use of blocked isocyanates permits the preparation of stable compositions regardless of the solvent and enables one to prepare fabrics having a propensity for subsequent durable setting.
several months after treatment without any further chemical treatment. For example, as further illustrated elsewhere in this application, a fabric can be converted into a garment and set in a given configuration by dry heating and pressing months after the fabric has been impregnated with the compositions of this invention containing a blocked polyisocyanate. Of course, the heating and pressing temperatures must be above the dissociation temperature of the blocked isocyanate.

‘A third essential ingredient in the compositions of the invention is a metal oxide such as zinc oxide, magnesium oxide e.g., magnesia), lithium red lead (lead tetraoxide), calcium oxide, iron oxide and titanium dioxide. The metal oxides perform three important functions in the compositions of the invention. They promote cross-linking or curing of the composition; they improve resistance to aging, heat, light and weather; and they further act as acid acceptors. The preferred metal oxide is zinc oxide although a combination of magnesium and zinc oxide is desirable and virtually necessary in compositions containing certain neoprenes, such as, for example, Neoprene-Type GN.

Generally, about 5 parts of metal oxide per 100 parts of neoprene provide satisfactory results although greater or smaller amounts may also be utilized. For example, in some instances, as much as 10 parts of the metal oxide may be incorporated, and higher amounts, from 10 to 15 parts, are often recommended in stocks requiring maximum heat resistance although the use of such large quantities results in products having somewhat reduced storage stability. Less than 5 parts per 100 is sometimes used in transparent products in order to reduce the pigmenting effect although such products should not be used in intimate contact with acid sensitive materials.

It is possible in some instances to obtain a significant amount of curing in the absence of a metal oxide if a zinc-containing accelerator is used. Examples of such accelerators include zinc salt of 2-mercaptobenzothiazole or zinc dibutyldithiocarbamate. Products utilizing these accelerators should, however, be used with caution since their aging resistance will be below that obtained from the metal oxides.

Although the compositions comprising a neoprene elastomer, a metal oxide, and a polyisocyanate or polyisothiocyanate may be applied as prepared to a fabric, it is often desirable that the composition be diluted with a liquid which is relatively inert to the compositions. That is, the compositions should be either prepared or added to a liquid to form solutions, dispersions or emulsions of the composition. The amount of liquid is not critical except that the viscosity of the solution or emulsion should be sufficiently low to permit the solution to impregnate the fabric. Maximum benefit is obtained when the composition of the invention impregnates the fabric as opposed to the deposition of a coating on the fibers of the fabric. For this reason, Brookfield viscosities of less than 5,000 centipoises are desirable and preferred.

The liquid utilized in the preparation of the solutions, dispersions, or emulsions of the invention may be either non-reactive organic solvents or in some instances, as explained further below, water. By “non-reactive” solvents as used herein is meant a solvent in which reactivity between the isocyanate and the neoprene or the fabric, even in the presence of a catalyst, is substantially inhibited.

Suitable organic solvents include halogenated hydrocarbons, such as trichloroethylene, methylene chloride, perchloroethylene, ethylene dichloride, and chloroform; aromatic solvents such as toluene, xylene, benzene and mixed aromatics such as the Solvesso types, n-butyl acetate, p-dioxane and methyl isobutyl ketone. Mixtures of such solvents may also be used.

The use of a nonreactive organic liquid permits the combination of all of the desired components of the composition in a single solution, emulsion or dispersion without any substantial reaction occurring among the components. In this manner, all of the components, including the cure accelerators are impregnated uniformly in the fabric in controllable amounts.

The use of water as a diluent or dispersing agent for the compositions of the invention is limited to those compositions wherein the isocyanate or isothiocyanate has been prereacted with one of the adduct-forming compounds described previously to form a blocked isocyanate or blocked polyisothiocyanate. Blocking of the reactive isocyanate groups is essential since these groups react readily with water if not blocked thereby eliminating their utility in the aqueous compositions of the invention. As mentioned previously, the compositions of this invention which contain the blocked isocyanates or isothiocyanates provide especially desirable results since these compositions may be impregnated into the fabric and dried without substantial reaction among the components of the composition and the fabric substrate. Reaction only occurs when the impregnated fabric is heated to a temperature which is sufficient to unblock or dissociate the blocked isocyanate which is then free to react. Until the impregnated fabric is heated to such a temperature, the fabric is in a presensitized state having a propensity for subsequent permanent pressing. The fabric presentsized in this manner is further characterized by excellent stability and shelf life, and the ability to be set in a given configuration in the dry state by heat alone. That is, the fabric need not be wet by water or any other liquid or reactant to develop the desirable permanent press characteristics.

Although the compositions described above are, in themselves, useful, they nevertheless are susceptible to improvement by the incorporation of other additives which impart properties desired for special applications and needs. Such additives include antioxidants, acrylic resins, polyhydroxy compounds, alkyd resins, thermosetting resins such as phenolic resins, melamine resins and urea formaldehyde resins, formaldehyde donors, silicones and wetting agents. The type, number and amount of these optional additives included in the compositions of the invention will depend upon the particular properties desired.

Neoprene-containing compositions have been found to age in the presence of air due to the slow attack by oxygen. Such aging results in a reduction of the desirable physical properties. This process is catalyzed further by heat and sunlight. Therefore, the use of efficient antioxidants is desirable if not essential in neoprene-containing compositions in order to assure outstanding resistance to aging. The antioxidants should be selected with care since they are not equally effective as antioxidants, and some have some undesirable secondary effects such as increased discoloration upon exposure to light or staining of the finishes. The more effective antioxidants generally cause discoloration or staining. The most commonly used antioxidants for black or dark-colored amine-type antioxidants such as those available under the trade name “Neozone”, Neozone A (N-phenyl-1-naphthylamine) and Neozone D (N-phenyl-2-naphthylamine), are examples of such amine antioxidants.

In light-colored or nonstaining neoprene-containing compositions, the use of noncoloring antioxidants is recommended. The substituted or hindered phenols are the most widely used antioxidants of this class and generally give good aging properties combined with maximum resistance to discoloration and staining. Examples of such hindered phenols include “Antioxidant 2246” which is 2,2’-methylene-bis(4-methyl-6-t-butylphenol) and “Antioxidant 425” which is 2,2’-methylene-bis(4-ethyl-6-t-butylphenol). Another phenolic nonstaining antioxidant available commercially is “San towthite” powder as a 40 percent solution in water.

Esters of unsaturated fatty acids have also been found to be useful to reduce the darkening of white and pastel colored neoprene-containing compositions. Principally, those oils of fatty acids having one or two double bonds are preferred. Examples of such oils include palm oil, predominantly palmitic acid, trilglyceride; olive oil, predominantly oleic acid trilglyceride; and safflower oil, predominantly linoleic acid trilglyceride. Combinations of these esters with the non-discoloring phenolic antioxidants have been found to be especially effective.
The incorporation of acid polymers into the compositions of this invention also provides a product having improved strength, and impregnated fabrics having improved crease retention quality. Acid polymers contemplated as being useful in this present invention are prepared from any of the polymerizable acids, i.e., those containing unsaturated groups. These polymers may be homopolymers of the acids or interpolymers of the acids and other monomers. Such acids, include, for example, acrylic acid, maleic acid, methacrylic acid and polymerizable phosphoric acids. Suitable monomers which may be copolymerized with the above acids include esters of the above acids such as ethyl acrylate and methyl methacrylate; alkyl fumarates and maleates; vinyl halides such as vinyl chloride; and other vinyl monomers such as styrene, acrylonitrile.

The acid polymers, as a general rule, are emulsion polymers containing varying amounts of solids, normally in the range of about 25 to 60 or 70 weight percent. Acrylic polymers of this type are readily available commercially (under the trade name Rhoplex), (under the trade name Hycar), (under the trade name Ucon).

The compositions of the invention may also contain polymeric polyhydroxy compounds. These polyhydroxy compounds may be water-soluble or insoluble. When incorporated into the compositions of the invention, they result in the formation of a fabric with improved flexibility and handle. By "polymeric polyhydroxy compound" is meant a linear long-chain polymer having terminal hydroxyl groups including branched, polyfunctional, polymeric polyhydroxy compounds as set forth below. Among the suitable polymeric polyhydroxy compounds there are included polyether polyls such as polyalkylene ether glycols, polyalkylene-arylene ether-thioether glycols and polyalkylene triols. Mixtures of these polyls may be used when desired.

The polyalkylene ether glycols may be represented by the formula HO(RO)nH, wherein R is an alkylene radical which need not be the same in each instance, and n is an integer. Examples of such glycols include polyethylene ether glycol and polypropylene glycol. Polyalkylene ether triols are obtained by reacting one or more alkylene oxides with one or more low-molecular weight aliphatic triols. The alkylene oxides most commonly used have molecular weights between about 44 and 250 and these include for example, ethylene oxide, propylene oxide, butylene oxide, and 1,2-epoxycyclohexane.

Representative examples of the polyalkylene ether triols include: polypropylene ether triol (MW 700) made by reacting 608 parts of 1,2-propylene oxide with 92 parts of glycerine; polypropylene ether triol (MW 1535) made by reacting 1,401 parts of 1,2-propylene oxide with 134 parts of trimethyl propane; and polypropylene ether triol (MW 6000) made by reacting 5,866 parts of 1,2-propylene oxide with 134 parts of 1,2,6-hexane triol.

Additional suitable polyols include polyoxypropylene triols; polyoxybutylene triols; Niax triol LG56, LG42, and LG112; T10L TG-400; and Actol 32-160.

Thermosetting resins may also be included in the compositions of this invention in amounts up to about 10 percent by weight. Examples of thermosetting resins which are useful include phenolic and aminoplast resins, such as the reaction products of phenol, cresol, xylol, urea, melamine and substituted melamines with aldehydes such as formaldehyde, furfuraldehyde, etc.

The term "phenolic resins" is used herein in its conventional meaning and includes the resins made from phenol and formaldehyde. Acid polymers contemplated as being useful in this composition are made by condensing an amine with an aldehyde such as by stirring one mole of urea with two moles of 37 percent formalin at 25-30°C. in alkaline solution until the aldehyde is completely reacted. The conditions for reacting melamine with aqueous formaldehyde are somewhat different from the reactions of urea. Because of the low solubility of melamine in water, the reactions are usually conducted at temperatures of 80°C-100°C to bring the melamine into solution more readily. The amino groups of melamine can each add two methylol groups, while in urea, apparently only one mole of formaldehyde adds to each amino group. Hexamethylen melamine is formed by heating melamine at 90°C with an excess of formaldehyde.

The alkyd resins are polymer resins obtained by reacting polyhydric alcohols such as glycols, glycerol, sorbitol, penterythritol etc., with polybasic acids such as phthalic acid, maleic acid, adipic acid, azelaic acid and sebacic acid. These resins may be modified with saturated and unsaturated monobasic acids, saturated and unsaturated monohydric alcohols, etc. The oil modified alkyd resins are polyester resins which have been modified with a drying and nondrying oil such as coconut oil, castor oil, soybean oil, linseed oil, tung oil and the acids and glycerides derived therefrom. Examples of such alkyd resins include coconut oil-modified glycerol phthalate containing about 33 percent by weight of fatty acids, and soybean oil-modified glycerol phthalate resins containing about 41 percent by weight of fatty acids. The alkyd resins are available commercially under a wide variety of trade names from a variety of sources. Water-soluble alkyd resins are available under the trade name AROTAP.

Typical aldehyde generating compounds which can be incorporated into the compositions of the invention include linear polymers, particularly those of the general formula CHO(CH2)nH which depolymerize to monomeric formaldehyde gas upon vaporization. In this class of compounds, there are included lower polyoxyethylene glycols, wherein n is from about 2 to 8; paraformaldehyde, wherein n ranges from about 6 to 100; alpha-polyoxy methylenes, wherein n is greater than about 100; and beta-polyoxy methylene wherein n is greater than about 100.

Polyoxyethylene glycol derivatives may also be utilized. Examples include the polyoxyethylene diamethacets and the lower polyoxyethylene dimethyl ethers. In general, higher temperatures, e.g., up to about 200°C are utilized to effect depolymerization of these derivatives. Formaldehyde acetals (formals) may also be utilized. Preferred formals are produced by reaction of formaldehyde with alcohols of the formula CH2(OH)(OR) in the presence of an acid catalyst, wherein R is alkyl or aryl alkyl. These compounds hydrolyze to formaldehyde and the parent alcohol. Preferred formals include methyl and 1,3-dioxolane.

Other suitable aldehyde generating compounds include the various methylol compounds, for example, methyl alkanolamine sulfites, including N-methyl-ethanolamine sulfite, methylamides such as N-methylformamide, N-methylacetamide and N-methyl acrylamide; and amines such as trimethylamine.

Also useful in the compositions of the invention are water-insoluble silicone fluids such as SF-350, a dimethyldisiloxane polymer. Other commercially available dialkyl polysiloxanes are useful.

The surfactants and wetting agents may be either nonionic or anionic. Examples of nonionic wetting agents include alkyl aryl polyether alcohols such as the ethylene oxide condensation products of octylphenol available under the trade name TRIFON X-100 and the ethylene oxide condensation products of nonylphenol and polyoxyethylene available under the trade name AEROSOL OT. Examples of anionic wetting agents include metallic salts of disproportionated resin acids and alkyl esters of sulphononic acids. Specific examples of the latter wetting agents include the dibenyl ester of sodium sulphononic acid available under the trade name AEROSOL NA, and the dimethyl ester available under the trade name AEROSOL MA.

Although small amounts, for example, from about 0.1 to about 10 percent by weight of the surfactant wetting agent have been found to be sufficient in the compositions of the in-
Composition A

<table>
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<tr>
<th>Parts by Weight</th>
<th>Composition</th>
<th>Neoprene 460</th>
<th>Bis phenol adduct of methylene bis(4-phenyl isocyanate), (40% in water)</th>
<th>Zinc oxide</th>
<th>Syn-Fac 905</th>
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<tr>
<td>100</td>
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<td>50</td>
<td>100</td>
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Composition B

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<th>Composition</th>
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<th>Bis phenol adduct of Composition A</th>
<th>Zinc oxide dispersion (50% in water)</th>
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<td>50</td>
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Composition C

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<th>Composition</th>
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<th>Lead tetoxide</th>
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<th>Neoprene L65 50% solids</th>
<th>Neozene D</th>
<th>Neozene D</th>
<th>Neoprene L65 50% solids</th>
<th>Zinc oxide dispersion (50% in water)</th>
<th>Neocot R</th>
<th>Neocot R</th>
<th>Nonionic surfactant</th>
<th>Water</th>
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Composition D

<table>
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<th>Composition</th>
<th>Neoprene L65 50% solids</th>
<th>Zinc oxide dispersion (50% in water)</th>
<th>Neoprene L65 50% solids</th>
<th>Bis phenol adduct of Composition A</th>
<th>Rophex E-358</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
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<td>50</td>
<td>50</td>
<td>15</td>
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</table>

Composition E

<table>
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<tr>
<th>Composition</th>
<th>Neoprene L65 50% solids</th>
<th>Zinc oxide dispersion (50% in water)</th>
<th>Neocot R</th>
<th>Neocot R</th>
<th>Nonionic surfactant</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>15</td>
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</tbody>
</table>

Composition F

<table>
<thead>
<tr>
<th>Composition</th>
<th>Neoprene L65 50% solids</th>
<th>Zinc oxide dispersion (50% in water)</th>
<th>Neoprene L65 50% solids</th>
<th>Bis phenol adduct of Composition A</th>
<th>Rophex E-358</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
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<td>50</td>
<td>50</td>
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</table>

Composition G

<table>
<thead>
<tr>
<th>Composition</th>
<th>Neoprene L65 50% solids</th>
<th>Bisphenol A (a phenolic non-staining antioxidant) (60% in water)</th>
<th>Neocot R</th>
<th>Neocot R</th>
<th>Nonionic surfactant</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
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Composition H

<table>
<thead>
<tr>
<th>Composition</th>
<th>Neoprene Type W</th>
<th>Zinc oxide</th>
<th>Magnesium oxide</th>
<th>Methylene bis(4-phenyl isocyanate)</th>
<th>Toluene</th>
<th>Neocot A (N-phenyl-isophthalic acid; 50% in water)</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>75</td>
<td>5</td>
<td>2</td>
<td>10</td>
<td>100</td>
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Composition I

<table>
<thead>
<tr>
<th>Composition</th>
<th>Neoprene L750 50% solids</th>
<th>Zinc oxide (50% in water)</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>30</td>
<td>30</td>
<td>30</td>
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</table>

Composition J

<table>
<thead>
<tr>
<th>Composition</th>
<th>Neoprene L65 50% solids</th>
<th>Neoprene L65 50% solids</th>
<th>Bis phenol adduct of Composition A</th>
<th>Nonic 218</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>15</td>
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<td>50</td>
<td>10</td>
<td>15</td>
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Composition K

<table>
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<th>Composition</th>
<th>Hydar 2671</th>
<th>Polyethylene Glycol 600</th>
<th>Water</th>
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<td>25</td>
<td>52</td>
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</tbody>
</table>

Composition L

<table>
<thead>
<tr>
<th>Composition</th>
<th>Hydar 2671</th>
<th>Ucon 50M8 2000</th>
<th>Syn-Fac 905</th>
<th>Rophex E-358</th>
<th>Water</th>
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</thead>
<tbody>
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<td>25</td>
<td>15</td>
<td>25</td>
<td>25</td>
<td>49.2</td>
<td>70</td>
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</tbody>
</table>

Composition M

<table>
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<tr>
<th>Composition</th>
<th>Trol G 4000</th>
<th>Rophex E-358</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>10</td>
<td>10</td>
<td>70</td>
</tr>
</tbody>
</table>

Composition N

<table>
<thead>
<tr>
<th>Composition</th>
<th>Trol G 4000</th>
<th>Rophex E-358</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>15</td>
<td>15</td>
<td>75</td>
</tr>
</tbody>
</table>

**In preparing the compositions which are dispersions or emulsions, it is often desirable to prepare dispersions of the separate ingredients which can then be mixed in the customary manner. It has been found, for example, that it is difficult to prepare a stable emulsion or dispersion containing the components when the components are indiscriminately added to water. On the other hand, mixing of emulsions of the individual components is accomplished readily. Thus, for example, it is preferred in preparing dispersions such as composition G to prepare a zinc oxide dispersion containing about 50 percent solids by stirring and mixing in a ball mill, 100 parts of zinc oxide in 35 parts of water, 20 parts of a 10 percent solution of Dextan 11, (a dispersing agent), 35 parts of a 10 percent solution of ammonium caseinate (a reacted casein), and 5 parts of a 10 percent solution of sodium silicate. This mixture is milled for about 24 hours to form a stable zinc oxide dispersion. The Sontowhole powder (40 percent solution) is likewise prepared in a ball mill by milling 100 parts of the Sontowhole powder in 80 parts of water, 30 parts of a 10 percent solution of Dextan 11, 30 parts of a 10 percent solution of ammonium caseinate and 10 parts of a 10 percent solution of sodium silicate. The bisphenol adduct dispersion (40 percent active ingredient) is prepared by the same procedure by milling 100 parts of the adduct with 80 parts of water, 30 parts of A 10 percent solution of the Dextan 11, 30 parts of a 10 percent solution of ammonium caseinate and 10 parts of a 5 percent solution of Aerosol OP, a wetting agent. After these separate emulsions are prepared, they are added to the Neoprene Latex 750 and Nonic 218 surfactant and mixed in the conventional manner. In the preferred practice of this invention, the textile fabric is impregnated with the compositions described above, either as solutions, dispersions or emulsions. The amount of solids contained in the solution, dispersion or emulsion preferably ranges from about 8 to 12 parts of the neoprene elastomer, from about 1 to 5 parts of the isocyanate and from about 0.2 to 1 part of the meta oxide, and the solids preferably should be diluted to provide a liquid having a sufficiently low viscosity to permit the composition to penetrate the fabric. Generally, the solution, dispersion or emulsion applied to the fabric will contain from about 40 to 50 percent by weight of solids.
The solutions, dispersions or emulsions are preferably padded on the fabric. Conventional padding or spraying equipment can be used for this purpose. Generally, a wet pickup of from about 60–100 percent based on the weight of the fabric is obtained providing for the incorporation of about 30–50 percent of the chemical solids.

After impregnation of the fabrics as described above, the fabrics are dried and then either stored or processed further. At this point, the impregnated fabrics have a propensity for subsequent durable setting which can be effected by heat curing in the dry state.

When the composition of the invention is prepared as an aqueous emulsion or dispersion and the diisocyanate is blocked such as by reaction with a phenol the fabric can be impregnated, dried and stored for a longer period of time since curing cannot be effected until the impregnated material is heated to a temperature sufficient to dissociate the blocked isocyanate. In most instances, the dissociation temperature is sufficiently high to provide a presensitized fabric which is stable over a wide range of temperatures. For example, the phenol adduct utilized in Composition G dissociates at a temperature of about 320°F, and therefore, the impregnated fabric must be heated at or above this temperature to effect the cure and set the fabric.

It is this latter aspect of the invention that provides the most desirable properties. The utilization of compositions such as Composition G permits the preparation of presensitized fabrics which can be cut, converted to garments and thereafter cured while being maintained in a preselected configuration to provide creases and pleats as desired. These creases and pleats have been found to possess improved wash and wear performance. That is, fabrics treated in this manner exhibit improved crease and shape retention and reduced shrinking when washed in a commercial washing machine. Such improved properties are imparted by the present invention on virtually all types of fabrics ranging from fabrics containing 100 percent natural fibers such as cotton or wool to fabrics composed exclusively of synthetic fibers, such as polyesters and polyamides. Fabrics containing blends of these fibers have also been found to be improved by the compositions of the invention. The compositions of the invention also have improved the performance of laminated fabrics, such as for example fabrics composed of two or more layers of fabrics or an outer layer of a fabric and an inner layer of a foam such as a urethane foam. Impregnation of such fabrics by the compositions of the invention does not effect the bond between the two layers, and can, therefore, be applied after lamination of the fabrics.

The following table illustrates the results obtained when a variety of fabrics is treated in accordance with the process of the invention. All the fabrics listed in the table, except fabric 4 were impregnated with the identified solutions to a wet pickup of 80 percent based on the weight of the fabric, dried, pressed on a Hoffman press utilizing a cycle of 40 seconds steam, 5 seconds bake and 5 seconds vacuum, and thereafter cured by heating to a temperature of 300°F for a period of 20 minutes. The treatment of fabric 4 differs in that the wet pickup of Composition K was 70 percent. The crease retention and flat dry results are based on a scale of from 1 to 5, 1 representing no crease retention and 5 representing an excellent crease. As can be seen from the data contained in the table, fabrics impregnated with the compositions of the invention exhibit excellent crease retention and flat dry properties and improve resistance to shrinkage.

<table>
<thead>
<tr>
<th>Fabrik</th>
<th>Composition</th>
<th>Crease retention</th>
<th>Flat dry performance</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Wool/nylon triacetate laminated</td>
<td>D</td>
<td>3.4</td>
<td>4.5</td>
</tr>
<tr>
<td>2. Wool/nylon triacetate laminated</td>
<td>G</td>
<td>3.2</td>
<td>4.5</td>
</tr>
<tr>
<td>3. 100% worsted wool</td>
<td>K</td>
<td>3.4</td>
<td>4.5</td>
</tr>
<tr>
<td>4. White shirt (100%) cotton</td>
<td>K</td>
<td>4.6</td>
<td>4.6</td>
</tr>
<tr>
<td>5. Same as 1</td>
<td>L</td>
<td>3.7</td>
<td>4.1</td>
</tr>
</tbody>
</table>

1 AATCC Test No. 98 C 194°F (overhead); after five, 194°F washes. 2 AATCC Test No. 98 C 194°F (overhead); after five, 194°F washes. 3 After 10 washes.

That which is claimed is:

1. A process for preparing garments having improved permanent press characteristics which comprises:
   a. treating a textile fabric with a composition comprising:
      i. a polychloroprene or a copolymer of chloroprene with acrylonitrile,
      ii. an isocyanate compound selected from the group consisting of polysiocyanates, polyisothiocyanates, adducts of said isocyanates with compounds possessing only one group containing a reactive hydrogen atom and mixtures of said isocyanates or adducts of isocyanates, and
      iii. a metal oxide selected from the group consisting of the oxides of the metal and iron, lead and tin;
   b. converting the treated fabric into a garment;
   c. maintaining the garment in a desired configuration; and
   d. subjecting the garment to a temperature sufficient to cure the treated fabric.

2. The process of claim 1 wherein the metal oxide is zinc oxide.

3. The process of claim 1 wherein the textile fabric is treated with an aqueous dispersion of the composition, and the isocyanate compound is an isocyanate adduct.

4. The process of claim 1 wherein the composition comprises from about 8 to 12 parts of polychloroprene, from about 1 to 5 parts of an isocyanate compound and from about 0.2 to 1 part of the metal oxide.

5. The process of claim 4 wherein the isocyanate is an alkyd diisocyanate.

6. The process of claim 1 wherein the textile fabric is a fabric containing at least some natural fibers.

7. A process for preparing garments having improved permanent press characteristics which comprises:
   a. impregnating a textile fabric with a composition comprising:
      i. a chloroprene polymer or a copolymer of chloroprene with acrylonitrile,
      ii. an adduct of a polysiocyanate with a compound having only one group containing a reactive hydrogen atom, and
      iii. a small amount of zinc oxide, magnesium oxide or calcium oxide;
   b. drying the treated fabric;
   c. converting the treated fabric into a garment;
   d. maintaining the garment in a desired configuration; and
   e. subjecting the garment to a temperature sufficient to cure the treated fabric.

8. The process of claim 7 wherein the polysiocyanate adduct is an aryl disiocyanate adduct.

9. The process of claim 7 wherein the metal oxide is zinc oxide.

10. The process of claim 7 wherein the composition also contains an antioxidant and an acrylic polymer.