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TREATMENT OF WOOL-CONTAINING TEXTILE MATERIALS

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This invention relates to the treatment of textile materials, and more particularly to the treatment of wool and wool-containing fabric and other textile materials to impart improved properties thereto, specifically increased resistance to shrinking. Still more particularly the invention is concerned with a process of reducing the shrinking tendencies of a wool-containing textile material which comprises preconditioning the said textile material by first treating it with an alkaline solution, specifically an alkaline aqueous solution or an alkaline organic solvent (e. g., an alkaline alcoholic) solution, of a peroxy compound (e. g., hydrogen peroxide, an organic or inorganic peroxide, a perborate, a persulfate, etc.) and thereafter removing the excess alkaline material, e. g., by washing the preconditioned textile. The pretreated textile, with or without being dried, is then impregnated with an impregnant comprising a curable (polymerizable) cationic aminoplast, more particularly a colloidal solution (specifically an aqueous colloidal solution) of a curable, cationic product of reaction of ingredients comprising an aminotriazine, e. g., melamine, and an aldehyde, e. g., formaldehyde. The colloidal solutions or dispersions of cationic aminoplasts used in practicing our invention are acidic, and the glass electrode pH value thereof may range, for example, between about 0.25 and about 5 or 6, or even as high as 6.8, when the cationic aminoplast (for instance, a partially polymerized, positively charged product of reaction of ingredients comprising an aminotriazine, e. g., melamine, and an aldehyde, e. g., formaldehyde) constitutes, on a net dry basis, about 6% by weight of the said solution. The cationic aminoplasts contained in the colloidal solutions are hydrophilic, and their degree of polymerization is less than that which characterizes gels and precipitates which are undispersible by agitation with water but sufficient to bring the particles thereof within the colloidal range.

Many different methods and agents have been used or suggested for use in the treatment of fabrics and other textiles composed of or containing wool in order to obviate or minimize their tendency to shrink on washing. The proposed treating agents have included halogens such as chlorine or bromine, solutions of sulfuryl chloride, hypochlorous acid, hpyochlorites (e. g.,

sodium hypochlorite), alone or in the presence of acidifying agents; alkalies (e. g., sodium or potassium hydroxide) or organic bases, such agents being used under conditions such that only the surface structure of the wool fibers is altered; enzymes such as papain; and reducing agents such as sodium hydrogen sulfide and mercaptoacetic (thioglycolic) acid. Another proposed shrinkage-control process has involved treating the wool-containing textile with an aqueous solution of a heat-curable resin, which is thereafter cured in situ, and then with a chlorine solution. Still another suggested process has involved first treating the woolen fabric with an alcoholic caustic solution in such a manner that there is no intrinsic damage to the fibers and no harmful effect on the hand of the fabric. In the second step of the process the woolen fabric is impregnated with an aqueous solution or dispersion of a heat-curable amino-aldehyde (e. g., urea-formaldehyde, melamine-formaldehyde, etc.) resin-forming material or precondensate, after which the impregnated fabric is heated to insolubilize the impregnant. (For a more complete description of this last-mentioned process, see Pfeffer, Jr. et al. Patent No. 2,395,791 wherein also are discussed the prior art processes described briefly above and why they are objectionable or fail completely to meet the requirements of a wool shrinkage-control agent or process.)

It was also suggested prior to our invention (see British Patent No. 553,923) that wool-containing textile materials be treated with an aqueous alkaline solution (pH of 8 to 12) of a substance capable of evolving oxygen (e. g., hydrogen peroxide, sodium peroxide, a persulfate, etc.) in order to reduce the tendency of the textile to felt and shrink. This treatment may be applied to the textile either before or after it has been treated with a sulfur-containing reducing agent, e. g., sulfur dioxide, a sulfite, a bisulfite, a hydrosulfite, etc. (see British Patent No. 579,584). Another proposed shrinkage-control treatment (see British Patent No. 564,958) has involved subjecting the wool-containing textile material to a mild chlorinating or brominating treatment controlled at a pH of 4 and then to treatment with an aqueous alkaline solution of hydrogen peroxide. Still another proposed method (see British Patent

No. 569,730) of imparting shrinkage-resistance characteristics to wool-containing textiles has involved treating the textile with an aqueous solution of a permanganate, e. g., potassium permanganate, and subsequently with either an aqueous solution or suspension of a nitrogen-chloro compound, e. g., nitrogen trichloride, monochloroamine, etc., or a dilute aqueous solution of an alkaline hypochlorite or hypobromite.

A process which is now widely used to render woolen and wool-containing textile materials resistant to shrinkage involves treating the textile with an aqueous solution or dispersion of an alkylated, specifically methylated, methylol melamine, followed by heating the impregnated material to cure the alkylated methylol melamine to a substantially water-insoluble condition. This method is described, for example, in Johnstone, Jr. et al. Reissue Patent No. 22,566.

It was also suggested prior to our invention that the fastness of dyed textile material, specifically cotton, mercerized cotton, ramie, jute, hemp, sisal, silk, artificial silk and wool could be improved by immersing the dyed material, immediately after the dyeing thereof, in an aqueous bath consisting of a solution of an aminotriazine-aldehyde resin, more particularly a melamine-formaldehyde resin, in a water-soluble stage, which solution has been subjected to the action of a strong mineral acid, specifically hydrochloric acid, for a period of time sufficient to convert the resin into substantive state. The bath contains the amount of resin which it is desired to apply to the textile material, and since the resin is substantive it is exhausted from the solution and deposited on and between the fibers of the textile material. The final step of the process is the drying of the wet impregnated textile.

The present invention is based on our discovery that a wool-containing textile material including wool itself in fabric or other form can be rendered highly resistant to felting and shrinking by means of a cationic aminoplast which is curable or polymerizable at normal or at an elevated temperature and which is applied in the form of a colloidal solution, more particularly an aqueous colloidal solution, provided that the wool-containing textile is preconditioned as described briefly in the first paragraph of this specification prior to treatment with the cationic aminoplast, which may be, for example, a cationic (positively charged) methylated or other alkylated methylol melamine. The instant invention is separate and patentably distinct from that disclosed and claimed in the copending application of Linton A. Fluck, Jr., John E. Lynn and Edward A. Schuman, Serial No. 43,963, filed concurrently herewith, and in which last-named application the disclosed and claimed invention involves an improvement in a method of reducing the shrinking tendencies of a wool-containing textile material with an aminoplast, more particularly a non-cationic aminoplast, adapted to effect this result, which improvement comprises treating the said textile material with an alkaline solution of a peroxy compound, e. g., hydrogen peroxide, prior to treatment with the said non-cationic aminoplast.

It was wholly unobvious and unexpected that pretreating a wool-containing textile with an alkaline solution of a peroxy compound would make it possible to use a cationic aminoplast as an agent for rendering such textiles highly shrink-resistant and that, as a result of such pretreatment, a substantially lesser amount of such an aminoplast can be employed as compared with a similar aminoplast which has not been rendered cationic and still obtain a desired degree of shrinkage resistance together with other advantages described below. The results are all the more surprising in view of our findings that the combination of treatments produces results quite different from either treatment alone, as shown by examples which follow, and results which are not merely the sum of the individual treatments.

Other advantages flowing from the peroxy pretreatment, and which also were quite surprising and in no way could have been predicted, include the general uniformity in the results obtained with a given cationic aminoplast in treating a wider variety of textiles composed of or containing a proteinaceous material, specifically wool; and the ability to impart shrink-resistant properties to woolen textiles which heretofore could not be rendered resistant to shrinking to an appreciable or a commercially satisfactory degree by previously known methods. Still another advantage accruing from our invention is that the applied cationic aminoplast can be dried and cured or polymerized, as by a condensation-polymerization reaction, to its ultimate form (in which form the cured or polymerized aminoplast is no longer dispersible in water to form a colloidal solution or dispersion) at a much lower temperature, for example at temperatures ranging from room temperature (65° F.–85° F.) to 225° F., as compared with the temperature required for similar aminoplasts which have not been rendered cationic, and still impart the same high order of shrink-resistant characteristics to the treated textile. Since the use of lower curing or polymerization temperatures provides a material reduction in the cost of treating the textile, it is immediately apparent that this advantage alone is of great practical significance and commercial importance.

The reasons why the treatment of a wool-containing textile with an alkaline solution of a peroxy compound prior to treatment with a cationic methylated methylol melamine or other cationic aminoplast is able to produce the valuable and unobvious results above described are not understood. One possible explanation, which is supported by our visual observations, is that the treatment in some way alters the physical and/or chemical characteristics of the wool so that better wetting and/or penetration occurs upon subsequent treatment with a cationic aminoplast. This improvement in wettability and/or penetrability is carried through any subsequent dyeing operation, as evidenced by the fact that no substantial differences in results are obtained when a dyeing step intervenes between the initial preconditioning treatment of the undyed wool-containing textile and the final treatment of the textile with a cationic aminoplast.

In carrying our invention into effect we first prepare an alkaline solution, preferably an aqueous alkaline solution, of a peroxy compound, more particularly such a compound which, in an aqueous medium, yields hydrogen peroxide or, ultimately, oxygen. Illustrative examples of peroxy compounds that may be employed are hydrogen peroxide and other inorganic peroxides (e. g., sodium, potassium and other alkali-metal peroxides), the organic peroxides (e. g., benzoyl peroxide, acetyl peroxide, lauroyl peroxide, succinyl peroxide, tert-butyl hydroperoxide, etc.), the various persalts including, for instance, the alkali-metal (e. g., sodium, potassium, etc.), ammonium and other perphosphates, persulfates, perborates, percarbonates, etc., these persalts in aqueous solutions forming hydrogen peroxide. Organic peracids (e. g., peracetic acid, perbenzoic acid, etc.) and the alkali-metal and ammonium salts of such acids also may be employed. Other percompounds which may be used include organic and organometallic salts of hydrogen peroxide, for instance tetramethyl, tetraethyl and other tetralkyl ammonium peroxides, triethyl tin peroxide, etc.

The concentration of peroxy compound in the alkaline solution may be varied widely depending, for example, upon the particular wool-containing textile employed, the particular peroxy compound used, the time and temperature of the treatment, etc. In general, however, the peroxy compound is used in an amount equivalent (oxidation equivalency) to at least about 0.05% by weight of hydrogen peroxide, based on the weight of the alkaline solution. Thus, when the peroxy compound is hydrogen peroxide, the alkaline pretreating solution ordinarily contains at least about 0.05%, more particularly at least about 0.1%, and preferably at least about 0.2%, by weight of hydrogen perixide; or, if the peroxy compound charged to the bath is other than hydrogen peroxide, then an amount thereof which is chemically equivalent as an oxidizing agent to at least about 0.05%, more particularly at least about 0.1% and preferably at least about 0.2%, by weight of hydrogen peroxide is employed. In most cases optimum results have been obtained when the concentration of the peroxy compound in the alkaline solution is equivalent to a hydrogen peroxide concentration of at least 0.3%, more particularly at least 0.5 or 0.6% by weight thereof. Higher concentrations of peroxy compound, as for example concentrations equivalent to hydrogen peroxide concentrations of the order of 2.5 or 3% or even 4 or 5% or more may be used if desired, but no particular advantages appear to accrue therefrom. Due to its relatively low cost and ease of handling the preferred peroxy compound is hydrogen peroxide, and generally it constitutes from about 0.3 to about 3%, more particularly from 0.5 to 2.5%, by weight of the alkaline solution, which is preferably an aqueous alkaline solution. Although the preferred alkaline solution of the peroxy compound is an alkaline solution of the said compound in water, other alkaline solutions in which the peroxy compound is soluble may be employed such, for example, as alkaline solutions of the peroxy compound in an alcohol (e. g., a monohydric alcohol such, for instance, as ethanol, propanol, butanol, etc.), or in a mixture of water and an alcohol.

If the peroxy compound employed is of a kind which does not yield an alkaline or a sufficiently alkaline treating bath, then the bath is made alkaline or is brought to a desired degree of alkalinity, as for example a pH of from about 8 to about 12, and more particularly to a pH of between 9 and 11, by the addition of an alkaline material or a material which reacts alkaline in aqueous solution. Any material capable of rendering the treating solution alkaline or of bringing the solution to a desired degree of alkalinity may be used, e. g., the sodium, potassium and other alkali-metal hydroxides, acetates, carbonates and bicarbonates, ammonium hydroxide, amines (e. g., triethanolamine), borax, guanidine carbonate, disodium phosphate, sodium silicates, soap, etc. Mixtures of substances which react basic in the aqueous or other solution of the peroxy compound may be used, if desired, for instance mixtures of such substances as those just mentioned by way of illustration.

The wool-containing textile to be preconditioned is treated with the alkaline solution of the peroxy compound in water or other solvent, preferably a volatile solvent, by any suitable method, for example by immersing the textile in the solution. The treatment may be effected by continuous, semi-continuous or batch methods, or by combinations of such methods. For instance, a wool-containing textile in skein, bolt or other form may be immersed in the treating solution for a period sufficient to effect the desired preconditioning treatment; or, the textile in, for example, the form of a woven fabric may be continuously passed through a bath of the solution at a rate such that it is immersed in the bath for a period sufficient to effect the desired results. If necessary, as for example when the wool-containing textile undergoing treatment contains free acid, additional basic material may be added to the pretreating bath during the treatment in order to maintain the bath at a desired pH value above 7.

The time of the treatment of the wool-containing textile with the preconditioning solution may be varied considerably. Ordinarily, however, the contact time is at least 15 minutes, although in some cases even shorter periods (for instance periods of the order of 5 or 10 minutes) may be effective. Better results generally are obtained when the textile is in intimate contact with the preconditioning solution for a period longer than 15 minutes, for example 30 minutes or longer. The time of contact may be 3 or 4 hours, or even as much as 1, 2 or more days in some cases, depending, for example, upon the particular wool-containing textile which is being pretreated, the pH and temperature of the treating solution, the kind and concentration of peroxy compound therein, and other influencing factors. In most cases, however, no particular advantages appear to accrue when the contact time is longer than about 30 minutes, such a contact time usually being as effective as a contact time of 60 minutes or even of 3 hours when the other conditions including the peroxy compound (e. g., hydrogen peroxide), concentration thereof, alkaline material (e. g., sodium silicate), pH of the solution (e. g., about 10), temperature of the solution, etc., are maintained substantially constant.

The temperature of the preconditioning solution also may be varied considerably but ordinarily it is within the range of about 110° F. to about 160° F., the optimum temperature usually being about 125° F. In some cases, however, the solution temperature may be lower than 110° F., e. g., between 70° or 80° F. and 105° F., or it may be higher than 160° F., e. g., 165° F. or 175° F.

After treating the wool-containing textile with the alkaline solution (preferably an aqueous alkaline solution) of the peroxy compound, the textile is water-washed and/or otherwise treated to remove the excess alkaline material. For example, the treated textile, after squeezing out the excess liquid, may be merely water-washed to remove the excess alkali; or, it may be water-washed and then rinsed in a dilute acid solution (e.g., a 0.2 to 6 or 8% solution of a mineral acid such, for instance, as phosphoric acid or sulfuric acid, or a 0.2 to 10% solution of an organic acid such, for example, as formic acid or acetic acid), after which the acid-rinsed textile is washed, e.g., with water, to remove the excess acid and water-soluble salts; or, the initial washing with water may be omitted, and merely a dilute acid rinse followed by a water wash may be used. The cycle of rinsing in dilute acid followed by water washing may be repeated numerous times, if necessary, in order to remove the excess alkaline material from the treated textile.

The preconditioning treatment herein described is applicable to both undyed and dyed wool-containing textiles. When applied to dyed wool-containing textiles there is usually some loss of dyestuff, but whether or not this occurs depends, of course, upon the resistance of the particular dyestuff to the alkaline solution of the peroxy compound.

The preconditioned textile may be dyed by any of the methods conventionally used in dyeing wool or wool-containing textiles without detracting from the benefits of the peroxy pretreatment. For instance, wool in yarn, skein, fabric or other form may be dyed by top-chrome, meta-chrome or bottom-chrome dyeing methods. Typcal of such dyeing processes are those briefly described below:

Top-chrome dyeing.—The dyestuff is applied first to the cloth in a boiling bath. The bath is cooled, a chrome salt (usually sodium or potassium dichromate) added, the bath brought to the boiling point, and then boiled for from 30 to 45 minutes.

Meta-chrome dyeing.—The chrome salt and dyestuff are applied to the wool in the same bath. Dyeing is started cold and finished at the boil.

Bottom-chrome dyeing.—Wool cloth is first boiled with a chromate in an acidified bath, the chrome color going onto the wool to give a dark green shade. The cloth is then boiled in a bath containing the dyestuff. The finished cloth may be any shade. Or, the wool may be dyed with a metallized dyestuff or an acid dyestuff such as are commonly used in dyeing wool and in accordance with conventional practice. The wool also may be dyed with vat dyes.

The final operation in carrying our invention into effect is the treatment of the preconditioned, dyed or undyed, wool-containing textile with a curable (polymerizable), cationic aminoplast adapted to render the textile resistant to shrinking when the said aminoplast has been sufficiently cured (polymerized) at normal or at an elevated temperature, more particularly a colloidal solution (e.g., an aqueous colloidal solution) of a curable (polymerizable), cationic product of reaction of ingredients comprising an aminotriazine, e.g., melamine, ammeline, guanamine, acetoguanamine, etc., and an aldehyde, e.g., formaldehyde. Also suitable for use in practicing our invention are curable, cationic products of reaction of ingredients comprising (1) an aminotriazine, e.g., melamine, (2) urea, or an alcohol, e.g., methanol, ethanol or other monohydric alcohol, or both urea and an alcohol, and (3) an aldehyde, e.g., formaldehyde. Ordinarily we use an aqueous colloidal solution of a partially polymerized, positively charged (cationic) product of reaction of ingredients comprising melamine and formaldehyde, which solution has a glass electrode pH value within the range of about 0.25 to about 4.0 when the said reaction product, on a net dry basis, constitutes about 6% by weight of the said colloidal solution. In this colloidal solution the degree of polymerization of the aforementioned positively charged product of reaction of ingredients comprising melamine and formaldehyde is less than that which characterizes gels and precipitates which are undispersible (that is, not permanently dispersible) by agitation with water but is sufficient to bring the particles thereof within the colloidal range. The colloidal solutions employed in the examples which follow have the aforementioned characteristics with respect to pH, degree of polymerization of the reaction product contained therein, etc.

The colloidal solutions of curable, cationic aminoplasts used in practicing our invention and the cationic aminoplastic themselves are prepared by known methods. Such methods are described in, for instance, Wohnsiedler et al. Patents 2,345,543 and 2,356,718, Pollard Patents 2,394,009, 2,417,014 and 2,423,428, and in the copending application of Henry P. Wohnsiedler and Walter M. Thomas, Serial No. 516,932, filed January 4, 1944, now Patent No. 2,485,079, and any of the cationic aminoplasts or colloidal solutions thereof disclosed (including those which are claimed per se or are a part of the claimed subject matter) in the aforementioned patents and application may be used in practicing our invention. Alkylated methylol melamines such as are disclosed in Johnstone, Jr. et al. Reissue Patent No. 22,566 may be rendered colloidal by treatment with an acid in a manner such as is described in the aforementioned Wohnsiedler et al. patents and application and also, for example, in the aforementioned Pollard patents.

As is now known, certain aminoplasts including aminotriazine-aldehyde condensation products, when prepared in the presence of definite quantities of free acid (e. g., hydrochloric, phosphoric, pyrophosphoric, nitric, formic, acetic, chloroacetic, etc.) or subsequently reacted with these same quantities of acid after their preparation by other methods, can be converted into colloidal dispersions wherein positive electrical charges are carried by the colloidal particles of the resin. In this condition the resin solutions or colloidal dispersions exhibit the property of migration of the resin particles toward the cathode upon electrophoresis of the dispersion, and this property characterizes all the aminoplast compositions employed in practicing the present invention. (The cataphoresis is preferably carried out by passing a direct current of 90–120 volts through platinum electrodes immersed in the colloidal solution.) In general, such aminoplast resins or condensation products, which may be designated more specifically as "acid-type" aminoplast resins or condensation products, and which include the acid-type aminotriazine-aldehyde condensation products, are readily identified by the following characteristics:

(1) When freshly prepared they form clear solutions in both water and acids; and

(2) The clear solutions are converted upon aging first to hydrophilic colloids, then to a water-dispersible gel stage, and finally upon drying to a water-insoluble resin.

Other and more specific characteristics of colloidal aqueous solutions of a partially polymerized, positively charged melamine-formaldehyde condensation product are disclosed and included in the claims of the aforementioned Wohnsiedler et al. Patent No. 2,345,543; and characteristics of colloidal aqueous solutions of a partially polymerized, positively charged guanamine-formaldehyde condensation product, in Wohnsiedler et al. Patent No. 2,356,718. As pointed out in these patents, the formation of a colloidal solution is shown in the first instance by the presence of a definite Tyndall effect under the influence of a beam of light or in the dark field microscope. Although the colloidal solution is infinitely dilutable with water it is extremely acid-sensitive, and this constitutes a very delicate test to determine whether any colloidal acid-type resin is present. When a strong acid such as 6N hydrochloric acid is added to a clear, acidified resin solution that has not been aged no immediate change occurs, but when the acid resin has been aged for a time sufficient to bring even a small part of the resin particles within the colloidal range these particles are precipitated by the addition of the acid. This reaction with strong acids can be used to estimate the degree of polymerization of the resin solution since the amount of acid necessary for precipitation becomes less with increasing age of the solution.

Illustrative examples of other curable cationic aminoplasts that may be employed in practicing the present invention are given, together with methods of preparing such aminoplasts and colloidal solutions containing the same, in the aforementioned Wohnsiedler et al. copending application Serial No. 516,932. The colloidal solutions disclosed and claimed in this application may be described more particularly as being a colloidal aqueous solution of a resinous copolymer (or intercondensation product) of melamine, urea and formaldehyde, which copolymer contains at least 0.7 mole of melamine for each 4 moles of urea and about 1 to 4 moles of combined formaldehyde for each mole of melamine plus urea and the said copolymer having a degree of polymerization such that the dispersed particles thereof are colloidal in character and size. In such colloidal aqueous solutions a positive electric charge is carried by the dispersed colloidal particles of the copolymer or intercondensation product as shown by their migration toward the cathode upon the passage of a direct current through the solution.

For a more complete understanding of the preparation of colloidal solutions containing curable cationic aminoplasts that may be used in practicing our invention, reference is made to the above-mentioned Wohnsiedler et al. copending application Serial No. 516,932 and the aforementioned Pollard Patents 2,394,009, 2,417,014 and 2,423,428 and Wohnsiedler et al. Patents 2,345,543 and 2,356,718, the cationic aminoplast compositions therein disclosed (including those which are claimed) being illustrative of those which can be employed in carrying the present invention into effect.

The curable cationic aminoplast may be applied to the wool-containing textile material in various ways. For example, if the cationic aminoplast is to be applied in the form of an aqueous colloidal solution containing the same, the dry or substantially dry textile to be treated may be immersed in the aqueous colloidal solution containing particles of cationic aminoplast of colloidal size, e. g., from 0.01 to 1 micron in diameter, and then passed through pressure rolls, mangles or centrifugal extractors to secure uniform impregnation and a controlled removal of the excess material. The woolen fabric or other wool-containing textile, however, also may be impregnated by other methods, e. g., by spraying.

The liquid composition (e. g., an aqueous liquid composition) containing the curable cationic aminoplast in the form of a colloid is diluted with a suitable diluent or dispersion medium (e. g., water), if necessary, to yield a finishing composition having the concentration of solids desired for textile application. Thus, the concentration of solids in the liquid composition may be, for instance, from about 1 to about 20% by weight of the composition depending, for example, upon the particular diluent or mixture of diluents employed, method of expression of the wet, impregnated textile, etc. Ordinarily, however, the curable cationic aminoplast constitutes from about 3 or 4% to about 10 or 12% by weight of the liquid (e. g., aqueous) finishing composition. The diluent or dispersion medium for the curable cationic aminoplast may be varied depending, for example, upon the characteristics of the said aminoplast, but in the case of the preferred cationic aminoplasts it is usually water; or it may be, for instance, a mixture of water and an alcohol, e. g., ethyl alcohol, glycerol, etc.

The pick-up of the liquid (e. g., aqueous) finishing composition by the wool-containing textile material should be adjusted so that the finished textile contains an amount of cured aminoplast sufficient to impart shrink-resistant characteristics thereto which at least are adequate to meet the minimum commercial requirements. This will vary somewhat depending, for instance, upon the particular wool-containing textile being treated, the particular preconditioning treatment which has been given to it, the particular aminoplast employed, the particular properties desired in the finished textile, etc. Ordinarily, however, the pick-up is adjusted so that the amount of aminoplast in the finished textile is not more than about ¾ (usually only about ½ or less) as much by weight as that required to obtain the same shrinkage-resistant characteristics in the absence of the preconditioning treatment. For example, the pick-up of the liquid finishing composition by the wool-containing textile material may be adjusted so that the finished textile contains from about 1 or 2% to about 6 or 8%, more particularly from about 3% to about 5%, by weight of the dry textile, of the cured (polymerized) aminoplast which, prior to curing is a curable (polymerizable) cationic (positively charged) aminoplast. In some cases, as for instance in the case of those woolen or wool-containing textiles which normally are not amenable to treatment with a cationic aminoplast alone to yield a commercially satisfactory shrink-resistant textile, the pick-up of the liquid finishing composition may be adjusted so that the finished textile contains as much as 10% or even about 15% or more, by weight of the dry textile, of the cured aminoplast. The amount of aqueous or other liquid finishing composition picked up by the textile, that is, the so-called "wet pick-up," may be varied as desired or as conditions may require and may range, for example, from about 30 or 40% up to about 150% or more by weight of the dry textile depending, for example, upon the particular textile treated, method and conditions of treatment, concentration of solids in the impregnating bath, amount of solids and properties desired in the finished textile, etc. When the woolen fabric or other wool-containing textile material is impregnated with 100%, by weight of the dry textile, of the textile-finishing composition, the impregnated textile will contain, after drying and curing of the curable cationic aminoplast, nearly the same percentage of cured aminoplast as the percentage of curable cationic aminoplast which is present in the textile-finishing composition.

The wet, impregnated woolen fabric or other wool-containing textile material is then dried and cured. The drying and curing temperatures may be varied considerably, but ordinarily temperatures within the range of about 65° or 70° F. up to about 225° or 250° F. are employed. In some cases drying and curing may be effected, if desired, at room temperature (65° F.–85° F.). The time of drying and curing may be shortened by increasing the temperature, for example to temperatures of the order of 180° F. to 212° F. The use of temperatures above 250° F. is not precluded, for example temperatures as high as 300° F. or 350° F. or even as high as 400 F. or more. As has been pointed out hereinbefore one of the main advantages accruing from the use of a curable cationic aminoplast as herein described is the lower drying and curing temperature that can be employed as compared with the temperature required for similar aminoplasts which have not been rendered cationic, and still impart the same high order of shrink-resistant characteristics to the treated textile. In all cases the drying and curing temperature and period are sufficient to dry the treated textile and to advance the cure or polymerization of the curable or polymerizable cationic aminoplast contained therein substantially beyond the stage which characterizes a gel, the cured or polymerized aminoplast being substantially water-insoluble and being incapable of yielding a stable, aqueous colloidal solution or dispersion.

The drying and curing operation is flexible and may be varied to suit the available equipment. Continuous methods are preferred. However, drying and curing also may be effected by framing the impregnated fabric to width, as on a pin tender, drying the framed fabric, batching it upon a shell and then allowing it to stand at normal or at an elevated temperature for a period sufficient to advance the cure or polymerization of the solid impregnant in situ to its ultimate form. In some cases drying and curing for from 2 hours to about 100 hours at temperatures within the range of about 65° F. to about 200° F. may be required. Heating the dried fabric at higher temperatures ranging from a few seconds to a few minutes at higher temperatures of the order of 212° F. to 250° F. or 300° F. or even as high as 400° F. or higher may be employed, the shorter periods being used at the higher temperatures.

After the woolen fabric or other wool-containing textile material has been treated as above described, it may be given a mild soaping treatment to render it softer and more pliable. It then may be given the usual finishing treatments such as decatizing, brushing, shearing, pressing, etc. Our process may be applied to wool and wool-containing textile materials, e. g., unwoven wool, yarns, worsteds, flannels, shirting, felts, knit or woven goods and others, and these may be composed of all wool or partly of wool and partly of another fibrous material, e. g., cotton, silk, any of the various rayons, any of the various other synthetic fibers including nylon, polyacrylonitrile fibers, fibers produced from casein, soya beans, etc.

In order that those skilled in the art better may understand how the present invention may be carried into effect, the following examples are given by way of illustration and not by way of limitation. All parts and percentages are by weight.

Example 1

The textile material used in this example was 100% all wool, women's wear flannel weighing 8 ounces per square yard. Pieces of this flannel, 9 inches by 23 inches in size, and which had been suitably marked for the purpose of measuring shrinkage after washing, were treated as described below:

A. A sample of the flannel was immersed for 30 minutes in an aqueous alkaline solution containing 0.6% by weight of hydrogen peroxide and which had been rendered alkaline to a pH of about 10 with sodium silicate. The solution was maintained at a temperature of about 125° F. during the treating period. The treated flannel was removed from the bath, rinsed first in water, then in a dilute (about 1%) aqueous solution of acetic acid and again in water, squeezed through a padder and then dried on a frame, while holding it to its original size of 9 inches by 23 inches, for about 6 to 10 minutes at about 225°–240° F.

B. Another sample of the flannel was impregnated by immersing it for at least about 1 minute in an aqueous colloidal solution containing about 6% by weight of the acetic acid colloid of methylated methylol melamine, more particularly methylated trimethylol melamine. The solution also contained 0.01% of a wetting agent, specifically a reaction product of an ethanolamide of a long chain fatty acid and ethylene oxide. The wet flannel was then squeezed through a padder to yield an impregnated cloth in which the wet pick-up was about 100% by weight of the dry flannel, after which the sample was framed to size as in A, and then heated for 10 minutes at 225° F. to dry the fabric and to advance the cure or polymerization of the colloidal methylated methylol melamine to a substantially completely cured or polymerized state, in which state it is substantially water-insoluble.

The acetic acid colloid of the methylated methylol melamine, more particularly methylated trimethylol melamine, was prepared as follows:

	Parts
Commercial methylated trimethylol melamine (about 80% solids, water constituting most of the remainder)	75
Water	883
Glacial acetic acid	42

were mixed together and then allowed to stand undisturbed at room temperature (about 65°–85° F.) for about 6 hours. The resulting aqueous colloidal solution contained about 6% by weight of the acetic acid colloid of methylated trimethylol melamine. The glass electrode pH value of the aqueous colloidal solution was 3.3.

C. A sample of the flannel was preconditioned as described under A, followed by treatment as described under B.

D. Same as C with the exception that the wet impregnated flannel was heated for 9 minutes at 290° F. to dry the fabric and to advance the cure of the acetic acid colloid of methylated methylol melamine as described under B.

After cooling to room temperature each of the samples of A, B, C, and D was removed from the frame and given 10 washings (soapings) of 10 minutes each at 100° F. in accordance with Federal Specifications CCC–T–191*a*. After steam pressing and conditioning for about 16 hours at 70° F. and 65% relative humidity, the samples were measured for shrinkage in inches per yard, which values were then changed to percentages. The warp shrinkage in per cent of each of the treated samples, together with that of the untreated sample which had been similarly washed, are shown below. Other shrinkage values given in other examples were determined in a similar manner.

Treatment	Warp Shrinkage in Per Cent
None	20.0
A	12.0
B	11.4
C	2.3
D	2.8

Example 2

The textile material used in this example was a Neolan dyed, 100% all wool flannel which weighed 8 ounces per square yard. It is typical of woolen fabrics which are less amenable to treatment with a methylated methylol melamine alone or with an acetic acid colloid of a methylated methylol melamine than the flannel used in Example 1. The treatments were as follows:

A. Same as A of Example 1.

B. Same as B of Example 1.

C. Same as C of Example 1.

D. Same as C of Example 1 with the exception that the aqueous colloidal solution of the acetic acid colloid of the methylated methylol melamine contained about 2% (instead of about 6% as in 1–C) of the said colloid.

E. Same as C of Example 1 with the exception that the aqueous colloidal solution of the acetic acid colloid of the methylated methylol melamine contained about 4% (instead of about 6% as in 1–C) of the said colloid.

The treated flannels and a control sample were washed as described under Example 1. The shrinkage data on the treated and washed samples are shown below, together with tensile strength data on the untreated sample and on the treated samples of A, B, and C.

Treatment	Warp Shrinkage in Per Cent	Tensile Strength in Pounds	
		Warp	Filling
None	40.0	31.5	26.5
A	20.6	33.0	32.0
B	23.3	39.5	31.0
C	2.2	45.5	32.5
D	3.0		
E	1.1		

The alkaline hydrogen peroxide pretreatment stripped the color from the yellow dyed wool used in this example but did not detract from the ability of the peroxide to precondition the cloth and thereby obtain outstanding and unobvious shrinkage-control characteristics upon subsequent treatment with the acetic acid colloid of the methylated methylol melamine.

Example 3

This example illustrates the results obtained when various temperature conditions are used in drying and curing the impregnated woolen textile. The woolen goods employed was a Neolan dyed woolen flannel such as was used in Example 2. The treatments were as follows:

A. Same as C of Example 1 with the exception that the dyed fabric, after being impregnated with the aqueous colloidal solution containing about 6% by weight of the acetic acid colloid of methylated methylol melamine, was dried and cured at room temperature (about 65°–85° F.) for about 16 hours.

B. Same as C of Example 1 wherein the impregnated textile was dried and cured for 10 minutes at 225° F.

C. Same as C of Example 1 with the exception that the temperature at which the impregnated textile was dried and cured for 10 minutes was 250° F.

D. Same as C of Example 1 with the exception that the impregnated textile was dried and cured for 9 minutes at 290° F.

The treated flannels and a control sample were washed as described under Example 1. The shrinkage data on the treated and washed samples are shown below:

Treatment	Warp Shrinkage in Per Cent
None	40.0
A	2.2
B	2.3
C	2.3
D	1.4

The peroxide pretreatment stripped the color from the wool as described under Example 2.

Example 4

This example illustrates the differences in the curing of polymerization conditions that can be used and the results obtained when a peroxide pretreated wool-containing textile is treated with an ordinary aqueous solution of a methylated methylol melamine and with aqueous colloidal solutions of an acid colloid, more particularly an acetic acid colloid, of the same methylated methylol melamine. The textile was an undyed women's wear flannel, 100% all wool, weighing 8 ounces per square yard. The treatments were as follows:

A. A sample of the flannel was preconditioned by treatment with an aqueous alkaline solution of hydrogen peroxide as described under A of Example 1. The preconditioned fabric was impregnated by immersing it for about 1 minute in an aqueous solution containing about 5% by weight of methylated methylol melamine, more particularly methylated trimethylol melamine. The solution also contained, by weight, 0.155% of a curing catalyst, specifically diammonium hydrogen phosphate, and 0.03% of a wetting agent, specifically dioctyl sodium sulfosuccinate. The wet flannel was then squeezed through a padder to yield an impregnated cloth in which the wet pick-up was about 100% by weight of the dry flannel, after which the sample was framed to size as in 1–A, and then heated for 10 minutes at 225° F. to dry the fabric and to advance the cure of the methylated methylol melamine as described under 1–B.

Another sample was treated as above described with the exception that the impregnated flannel was heated for 9 minutes at 290° F.

B. Same as A of this example with the exception that the preconditioned flannel was impregnated with an aqueous colloidal solution of an acetic acid colloid of the same methylated methylol melamine used in A; also, the aqueous colloidal solution contained about 4% by weight of the said colloid instead of about 5% of solids as in A of this example, and 0.01% of a non-ionic wetting agent, more particularly a reaction product of an ethanolamide of a long chain fatty acid and ethylene oxide. The wet impregnated textile was heated for 10 minutes at 225° F. as described in the first paragraph of A of the present example.

Other samples were treated as above described with the exception that each of the impregnated fabrics was heated under different conditions, viz., 10 minutes at 250° F. and 275° F., and 9 minutes at 290° F.

C. Same as B with the exception that samples of the woolen flannel were impregnated with the same acetic acid colloid in a concentration of about 6% by weight of the aqueous colloidal solution instead of about 4%. Each of the impregnated flannels was heated under different conditions, viz., 30 minutes at 180° F., 10 minutes at 225° F. and 9 minutes at 290° F.

The treated flannels and a control sample were washed as described under Example 1. The shrinkage data on the treated and washed samples are shown below:

Treatment	Warp Shrinkage in Per Cent, Sample Dried and Cured at—				
	180° F.	225° F.	250° F.	275° F.	290° F.
None	20.6				
A		13.3			3.9
B		5.3	3.6	3.9	3.9
C	5.0	3.3			2.8

When undyed wool-containing textile materials are to be rendered resistant to shrinking in accordance with our invention, a preferred preconditioning treatment includes the step of rinsing the peroxy pretreated textile with a dilute acid solution as hereinbefore described, followed by a water rinse. Under the usual conditions prevailing in a textile mill wherein the acid colloid is applied to a dyed textile, such an acid rinse is unnecessary since the wool is generally dyed in an acid (e. g., sulfuric or acetic acid) medium.

The following example illustrates the results obtained using a peroxy pretreating operation wherein, in one case, the peroxy-pretreated fabric was rinsed in a dilute acid solution and, in another case, the acid rinse was omitted.

Example 5

The woolen material used in this example was the same as that employed in Example 1. The treatments were as follows:

A. Same as C of Example 1.

B. Same as C of Example 1 with the exception that no acid rinse was employed in the preconditioning treatment. In other words, the treated flannel after removal from the alkaline hydrogen peroxide bath was merely rinsed in water prior to drying.

The treated flannels were washed as described under Example 1. The shrinkage data on the treated and washed samples are shown below:

Treatment	Warp Shrinkage in Per Cent
A	2.2
B	3.3

It is not necessary that the wool-containing textile material be dried after completion of the alkaline peroxy pretreatment. In other words the pretreated, washed and squeezed, impregnated textile may be treated while still wet with the aqueous colloidal solution of the acid colloid. This is shown by the following example.

Example 6

The textile material used in this was a dyed woolen flannel such as was used in Example 2. The treatments were as follows:

A. Same as D of Example 2 with the exception that the woolen fabric, after treatment with the aqueous alkaline solution containing 0.6% by weight of hydrogen peroxide and subsequent rinsing as therein described, was immersed directly, without being dried, in the aqueous colloidal solution containing about 2% by weight of the acetic acid colloid of methylated methylol melamine.

B. Same as A of this example with the exception that the aqueous colloidal solution contained about 4% by weight of the actetic acid colloid.

C. Same as A of this example with the exception that the aqueous colloidal solution contained about 6% by weight of the acetic acid colloid.

The treated flannels and a control sample were washed as described under Example 1. The shrinkage data on the treated and washed samples are shown below:

Treatment	Warp Shrinkage in Per Cent
None	40.0
A	3.3
B	1.9
C	1.4

It will be noted that the shrink-resistant properties of the treated flannels are of the same order as those of the treated fabrics of C, D, and E of Example 2.

Example 7

This example illustrates the preparation and use, in accordance with our invention, of an aqueous colloidal solution containing a hydrochloric acid colloid of methylated methylol melamine, more particularly methylated trimethylol melamine.

	Parts	Approx. Molar Ratio
Commercial methylated trimethylol melamine (about 80% solids, water constituting most of the remainder)	37.0	1.5
Aqueous hydrochloric acid (about 38% HCl)	7.5	1.0
Water	177.0	

were mixed together and then allowed to stand undisturbed at room temperature (about 65° F.–85° F.) for about 2 hours. The resulting aqueous colloidal solution contained about 13.3% of solids. It was diluted with water to yield a solution containing about 6% of solids, more particularly the hydrochloric acid colloid of methylated trimethylol melamine. The glass electrode pH value of the aqueous colloidal solution containing 6% solids was 2.3. This aqueous colloidal solution was used in rendering a wool-containing textile material resistant to shrinking as described below. The woolen material was a women's wear flannel such as used in Example 1.

A. Same a C of Example 1 with the exception that the above-described aqueous colloidal solution containing the hydrochloric acid colloid of methylated trimethylol melamine was used instead of the corresponding acetic acid colloid. The wet, impregnated fabric was dried and cured for 10 minutes at 225° F. Another alkaline hydrogen peroxide pretreated sample (B) was dried and cured for 30 minutes at 180° F., while a third, similarly pretreated sample (C) was dried and cured for 9 minutes at 290° F.

The treated samples and a control sample were washed as described under Example 1. The shrinkage data on the treated and washed samples are shown below, together with tensile strength data on the untreated sample and on the treated samples of A and C.

Treatment	Warp Shrinkage in Per Cent	Tensile Strength in Pounds	
		Warp	Filling
None	20.0	34.0	27.5
A (dried and cured at 225° F.)	4.2	36.5	21.5
B (dried and cured at 180° F.)	5.0	------	------
C (dried and cured at 290° F.)	4.2	38.0	30.0

Example 8

This example illustrates the preparation and use, in accordance with our invention, of an aqueous colloidal solution containing a phosphoric acid colloid of methylated methylol melamine, more particularly methylated trimethylol melamine.

	Parts	Approx. Molar Ratio
Commercial methylated trimethylol melamine (about 80% solids, water constituted most of the remainder)	25	1.0
Phosphoric acid (85% H_3PO_4)	13	1.4
Water	162	------

were mixed together and then allowed to stand undisturbed at room temperature (about 65° F.–85° F.) for about 10 hours. The resulting aqueous colloidal solution contained about 10% of solids, which thereafter was diluted with water to a solution containing about 6% of solids, and used as described in the preceding example instead of the hydrochloric acid colloid of that example. The glass electrode pH value of the aqueous colloidal solution containing 6% solids was 1.5. One wet, impregnated sample (A) was dried and cured for 10 minutes at 225° F., while another sample (B) was dried and cured for 9 minutes at 290° F. After being washed ten times as described under Example 1, the warp shrinkage in percent of each of the treated samples was 4.7. The warp shrinkage in per cent of a control (untreated) sample, after having been similarly washed ten times, was 20.0.

Example 9

This example illustrates the preparation and use, in accordance with our invention, of an aqueous colloidal solution containing a hydrochloric acid colloid of a melamine-formaldehyde reaction product, more particularly trimethylol melamine.

	Parts	Approx. Molar Ratio
Trimethylol melamine	100	1
Aqueous hydrochloric acid (about 38% HCl)	34	2
Water	736	------

were mixed together and then allowed to stand undisturbed at room temperature (about 65°–85° F.) for about 8 hours. The resulting aqueous colloidal solution contained about 11.5% of solids. One part was diluted with water to yield a colloidal solution (A) containing about 2% of a hydrochloric acid colloid of trimethylol melamine. Similarly, water was added to another part to form a colloidal solution (B) containing about 4% of the said colloid. Both solutions were applied to an alkaline hydrogen peroxide preconditioned woolen fabric as described under Example 8. The wet, impregnated fabrics were dried and cured for 9 minutes at 290° F. After being washed ten times as described under Example 1, the warp shrinkage in per cent of the fabric which had been treated with the colloidal solution (A) containing 2% of the colloid was 4.2, while that of the fabric which had been treated with the colloidal solution (B) containing 4% of the colloid was 5.6. The warp shrinkage in per cent of a control sample, after having been similarly washed ten times, was 20.0.

Example 10

The process of our invention is applicable to a wide variety of woolen textile materials, as shown by the following results of treating six different woolen fabrics. The treatments were as follows:

A. Same as C of Example 1.

B. Same as D of Example 1.

(The treatments of A and B involved a pretreatment of the fabrics with an aqueous alkaline solution containing 0.6% hydrogen peroxide and a finishing treatment with an aqueous colloidal solution containing about 6% of the acetic acid colloid of methylated trimethylol melamine. In the A treatment the wet, impregnated textile was dried and cured for 10 minutes at 225° F., while in the B treatment it was dried and cured for 9 minutes at 290° F.) The warp shrinkage in per cent of the untreated and treated fabrics, after being washed ten times as described under Example 1, are shown below:

Treatment	Warp Shrinkage in Per Cent of Woolen Fabrics					
	1	2	3	4	5	6
None	20.0	45.0	43.9	20.9	18.3	20.9
A	3.3	2.2	1.7	3.3	1.4	3.6
B	2.8	1.9	1.1	2.8	2.2	1.0

NOTE.—Fabrics 1, 2 and 3 were undyed women's wear flannel, 100% all wool, weighing 8 ounces per square yard. Fabrics 4 and 5 were Palatine dyed and top-chrome dyed, respectively, sulfuric acid carbonized woolen fabrics (100% all wool), weighing about 12 ounces for square yard, while Fabric 6 was an undyed, all wool, sulfuric acid carbonized fabric which also weighed about 12 ounces per square yard.

Optimum results in practicing our invention are obtained by using a peroxy pretreating solution which not only is distinctly alkaline when the wool-containing textile material initially is immersed therein but which is maintained alkaline during the entire period the woolen material is in the pretreating bath. This is shown by the following example.

Example 11

The general method of treatment was the same as described under C of Example 1.

Treatment	pH of Pretreating Solution		Warp Shrinkage in Per Cent After 10 Washes
	At Beginning of Pretreatment	At End of Pretreatment	
A	9.4	6	8.9
B	9.7	9	5.8

The woolen fabrics treated in A and B were sulfuric acid carbonized woolen materials and contained excess sulfuric acid. In A the pH of the pretreating solution was not controlled, while in B additional sodium silicate was added to the hydrogen peroxide pretreating solution during the treating period so that the pH of the solution did not fall below 9.

Example 12

The woolen fabric which was treated in this example was a Neolan dyed, 100% all wool flannel which weighed 8 ounces per square yard. Instead of using hydrogen peroxide in the pretreating bath as in the previous examples, a different peroxy compound, viz., sodium perborate, was employed. The treatments were as follows:

A. The woolen fabric was preconditioned by immersing it in an aqueous alkaline solution containing 1% by weight of sodium perborate. No sodium silicate was added to the bath since the sodium perborate alone brought the bath to a pH of about 10. The flannel was allowed to remain in the bath for 30 minutes while maintaining the solution at 125° F. The treated flannel was removed from the bath, rinsed in water to remove the excess alkali, squeezed through a padder and then dried on a frame, while holding it to its original size of 9 inches by 23 inches, for 10 minutes at 240° F. The dried flannel was impregnated by immersing it for at least about 1 minute in a textile-finishing composition comprising an aqueous solution containing about 5% by weight of methylated methylol melamine, more particularly methylated trimethylol melamine. The solution also contained, by weight, 0.155% of a curing catalyst, specifically diammonium hydrogen phosphate, and 0.03% of a wetting agent, specifically dioctyl sodium sulfosuccinate. The wet flannel was then squeezed and framed as described under B of Example 1, after which it was heated for 9 minutes at 290° F. to dry the fabric and to advance the cure of the methylated methylol melamine as described under 1B.

B. Same as A of this example with the exception that the textile-finishing composition comprised an aqueous colloidal solution containing 5% of an acetic acid colloid (prepared in a manner similar to that described under 1-B) of methylated trimethylol melamine. The solution contained no added curing catalyst, but did contain 0.01% of a non-ionic wetting agent, more particularly a reaction product of an ethanolamide of a long chain fatty acid and ethylene oxide.

The treated flannels and a control sample (untreated flannel) were subjected to washing in soap solution, more particularly five washing cycles of 10 minutes each followed by drying, and then were washed for 1 hour followed by drying. The data on shrinkage of the treated and washed samples are shown below:

Treatment	Warp Shrinkage in Per Cent
None	44.0
A	13.6
B	4.2

Example 13

The woolen fabric treated in this example was a women's wear flannel such as was used in Example 1. The treatments were as follows:

A. The flannel, without any preconditioning treatment, was treated with an aqueous colloidal solution containing 5% of the acetic acid colloid of methylated trimethylol melamine and 0.01% of a non-ionic wetting agent as described under A of Example 12.

B. Same as A of this example with the exception that the aqueous colloidal solution contained 10% of the acetic acid colloid.

C. Same as B of Example 12 with the exception that the aqueous alkaline pretreating solution contained 0.6% hydrogen peroxide and had been rendered alkaline to a pH of about 10 with sodium silicate. The textile-finishing composition comprised an aqueous colloidal solution containing 5% by weight thereof of an acetic acid colloid of methylated trimethylol melamine.

D. Same as C of this example with the exception that the aqueous colloidal solution contained 10% of the acetic acid colloid of methylated trimethylol melamine.

The treated flannels and a control sample were washed as described under Example 12. The data on shrinkage of the treated and washed samples are shown below:

Treatment	Warp Shrinkage in Per Cent	
	After 5 Washing Cycles of 10 Minutes Each	After 1 Hour of Additional Washing
None	18.1	21.1
A	7.2	8.3
B	7.4	5.8
C	1.4	1.9
D	1.4	1.7

Example 14

The same woolen fabric was treated as was used in Example 13. The treatments were as follows:

A. Same as A of Example 12 with the exception that the aqueous alkaline pretreating solution contained 0.6% hydrogen peroxide and had been rendered alkaline to a pH of about 10 with sodium silicate; also, the textile finishing composition comprised an aqueous solution containing only about 2½% of methylated trimethylol melamine, about 0.08% of diammonium hydrogen phosphate and about 0.03% of dioctyl sodium sulfosuccinate as a wetting agent.

B. The pretreating solution and method were the same as A of this example, while the finishing treatment also was the same with the exception that the textile finishing composition contained

2½% of an acetic acid colloid of methylated trimethylol melamine (see 1–B for method of preparation) and the same kind and amount of wetting agent as described under B of Example 12.

The treated samples and a control sample were washed as described under Example 12. The shrinkage data on the treated and washed samples are shown below:

Treatment	Warp Shrinkage in Per Cent [1]
None	21.1
A	5.8
B	3.3

[1] After 5 Washing Cycles of 10 Minutes Each Followed by 1 Hour of Additional Washing.

Example 15

This example illustrates the use of various preconditioning bath temperatures and of varying periods of immersion of the woolen material therein. The wool-containing textile material which was treated in this example was a 100% all wool, women's wear flannel such as was used in Example 1. The treatments were as follows:

A. Same as A of Example 12 with the exception that the aqueous alkaline pretreating solution contained 0.6% hydrogen peroxide and had been rendered alkaline to a pH of about 10 with sodium silicate; also, the preconditioning bath was maintained at a temperature of 100° F. for 8 hours instead of at 125° F. for 30 minutes as in 12–A. The textile-finishing composition contained about 5% by weight of methylated trimethylol melamine and the same kind and amount of curing catalyst and wetting agent as in 12–A.

B. Same as A of this example with the exception that the textile-finishing composition comprised an aqueous colloidal solution containing 6% of an acetic acid colloid (prepared in a manner similar to that described under 1–B) of methylated trimethylol melamine. The solution also contained 0.01% of a non-ionic wetting agent such as described under 1–B.

C. Same as A of this example with the exception that the alkaline hydrogen peroxide pretreating bath was maintained at a temperature of about 80° F. for about 24 hours.

D. Same as B of this example with the exception that the preconditioning solution was maintained at about 80° F. for about 24 hours.

The treated flannels and a control sample were washed as described under Example 12. The shrinkage data on the treated and washed samples are shown below:

Treatment	Warp Shrinkage in Per Cent [1]
None	20.0
A	4.4
B	2.5
C	4.4
D	1.7

[1] After 5 Washing Cycles of 10 Minutes Each Followed by 1 Hour of Additional Washing.

Example 16

This example illustrates the use of other peroxy compounds, specifically sodium peroxide and potassium persulfate, as components of the preconditioning bath. The woolen fabric was the same as that used in Example 1. The treatments were as follows:

A. Same as D of Example 1 with the exception that the pretreating bath contained ½% by weight of sodium peroxide. The sodium peroxide alone brought the solution to a pH of about 10 and no other alkaline material was added.

B. Same as D of Example 1 with the exception that the preconditioning solution contained 1% by weight of potassium persulfate. Sodium silicate was added in an amount sufficient to bring the pretreating bath to a pH of about 10.

The treated flannels and a control sample were washed as described under Example 12. The shrinkage data on the treated and washed samples are shown below:

Treatment	Warp Shrinkage in Per Cent	
	After 5 Washing Cycles of 10 Minutes Each	After 1 Hour of Additional Washing
None	----------	20.0
A	1.4	1.4
B	6.0	5.6

Example 17

The textile material used in this example was 100% all wool, undyed women's wear flannel similar to that used in Example 1. The treatments were as follows:

A. Same as C of Example 1, which involved pretreatment of the fabric with an aqueous alkaline solution containing 0.6% by weight of hydrogen peroxide and rendered alkaline with sodium silicate to a pH of about 10, and subsequent treatment of the dried, undyed fabric with an aqueous colloidal solution containing 6% by weight of an acid colloid of methylated trimethylol melamine.

B. Same as A of this example with the exception that after preconditioning the undyed fabric as in A but omitting the acetic acid rinse, it was dyed as follows: The dried, pretreated cloth was immersed for 1 hour in a dye bath maintained at boiling temperature and containing 3% of a dye (Calcocid Violet 4 BX, Color Index No. 698), 4% of acetic acid (approx. 28% CH_3COOH) and 7% of Glauber's salt, the percentages of dye, acetic acid and Glauber's salt being by weight and being based on the weight of the dry fabric. After rinsing the dyed textile in water, followed by drying, the dyed and dried fabric was treated with an aqueous colloidal solution containing 6% by weight of an acetic acid colloid of methylated trimethylol melamine as in A of this example.

The treated flannels were washed as described under Example 1, with the exception that each was given only 5 washings of 10 minutes each. The shrinkage data on the treated and washed samples are shown below:

Treatment	Warp Shrinkage in Per Cent [1]
A	5.8
B	5.6

[1] After 5 Washing Cycles of 10 Minutes Each.

It will be understood, of course, by those skilled in the art that our invention is not limited to the particular preconditioning compositions, curable cationic aminoplasts and conditions of treating a wool-containing textile with these compositions as has been given by way of illustration in the above examples, since the pretreating solution, the final textile-finishing composition and the application conditions may be widely varied as will be apparent from the description in this specification prior to the examples.

The terms "textile" and "textile material" as used generally herein and in the appended claims include within their meanings filaments, fibers, rovings, slivers, threads, yarns, twisted yarns, etc., as such or in woven felted or otherwise formed fabrics, sheets, cloths and the like.

We claim:

1. A process of reducing the shrinking tendencies of a wool-containing textile material which comprises preconditioning the said textile material by first treating it with an alkaline solution of a peroxy compound and thereafter removing the excess alkaline material, impregnating the preconditioned textile with an impregnant comprising a colloidal solution of a curable cationic aminoplast, and advancing the cure of the said aminoplast in situ, the amount of the cured aminoplast with which the said textile material is impregnated being sufficient to render it resistant to shrinking.

2. A process as in claim 1 wherein the alkaline solution of the peroxy compound is within a pH range of 8 to 12.

3. A process as in claim 1 wherein the impregnant comprises a colloidal solution of a curable, cationic product of reaction of ingredients comprising melamine and formaldehyde.

4. A process of reducing the shrinking tendencies of a dyed woolen fabric which comprises preconditioning the undyed fabric by first treating it with an aqueous alkaline solution of hydrogen peroxide and thereafter removing the excess alkaline material, dyeing the preconditioned fabric, impregnating the dyed fabric with an aqueous colloidal solution of a partially polymerized, positively charged product of reaction of ingredients comprising melamine and formaldehyde, said colloidal solution having a glass electrode pH value within the range of 0.25 to 6.8 when the said reaction product, on a net dry basis, constitutes about 6% by weight of the said colloidal solution, and the degree of polymerization of the said reaction product being less than that which characterizes gels and precipitates which are undispersible by agitation with water but sufficient to bring the particles thereof within the colloidal range, and polymerizing the said reaction product in situ to its ultimate form, the polymerized reaction product being substantially water-insoluble, and the amount of the said reaction product in polymerized state with which the fabric is impregnated being from 1 to 15% by weight of the dry fabric.

5. A process of reducing the shrinking tendencies of a dyed, wool-containing textile which comprises immersing the undyed textile in an aqueous solution rendered alkaline to a pH of between 8 and 12 and containing hydrogen peroxide in an amount corresponding to from 0.05% to 3% by weight of the said solution and thereafter removing the excess alkaline material thereby to precondition the undyed textile, dyeing the preconditioned textile, impregnating the dyed textile with an aqueous colloidal solution of a partially polymerized, positively charged alkylated methylol melamine, said colloidal solution having a glass electrode pH value within the range of about 0.25 to about 4.0 when the said alkylated methylol melamine, on a net dry basis, constitutes about 6% by weight of the said colloidal solution, and the degree of polymerization of the said alkylated methylol melamine being less than that which characterizes gels and precipitates which are undispersible by agitation with water but sufficient to bring the particles thereof within the colloidal range, drying the wet impregnated textile and polymerizing the said alkylated methylol melamine contained in the impregnated textile to its ultimate degree of polymerization, said drying and polymerization being effected at a temperature not exceeding 400° F., and the amount of the said alkylated methylol melamine in polymerized state with which the textile is impregnated being from 1 to 15% by weight of the dry textile.

6. A process as in claim 5 wherein the colloidal solution contains methylated methylol melamine rendered colloidal with acetic acid.

7. A process as in claim 5 wherein the alkylated methylol melamine is methylated methylol melamine, and the wet impregnated textile is dried and the methylated methylol melamine contained in the impregnated textile is polymerized to its ultimate degree of polymerization at a temperature within the range of about 65° F. to about 300° F.

8. The method of reducing the shrinking tendencies of a woolen textile material which comprises immersing the said material in undyed state for from ¼ to 3 hours in an aqueous solution rendered alkaline to a pH of between 9 and 11 and containing hydrogen peroxide in an amount corresponding to from 0.5 to 2.5% by weight of the said solution thereby to precondition the said textile material, said solution being maintained at a temperature of from 70° F. to 160° F.; removing the excess alkaline material from the preconditioned textile; impregnating the preconditioned textile from which the excess alkaline material has been removed with an aqueous colloidal solution of a partially polymerized, positively charged methylated methylol melamine rendered colloidal with acetic acid, said solution containing from about 2% to about 12% by weight of the said methylated methylol melamine and having a glass electrode pH value within the range of about 0.25 to about 4.0 when the said methylated methylol melamine, on a net dry basis, constitutes about 6% by weight of the said colloidal solution, and the degree of polymerization of the said methylated methylol melamine being less than that which characterizes gels and precipitates which are undispersible by agitation with water but sufficient to bring the particles thereof within the colloidal range; adjusting the pick-up of the said aqueous colloidal solution by the said preconditioned textile so that the amount of cured methylated methylol melamine in the finished textile is not more than about three-fourths as much by weight as that required to obtain the same shrinkproofing characteristics in the absence of the aforedescribed preconditioning treatment; drying the wet impregnated textile and polymerizing the said methylated methylol melamine contained in the impregnated textile to its ultimate degree of polymerization, said drying and polymerization

being effected at a temperature within the range of about 65° F. to about 225° F.

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