Patented Jan. 23, 1951

2,539,365

UNITED STATES PATENT OFFICE

2,539,365

TREATMENT OF WOOL-CONTAINING TEXTILE MATERIALS

Linton A. Fluck, Jr., Pluckemin, N. J., and John E. Lynn, Old Greenwich, and Edward A. Schuman, Stamford, Conn., assignors to American Cyanamid Company, New York, N. Y., a corporation of Maine

No Drawing. Application August 12, 1948,
Serial No. 43,963

15 Claims. (Cl. 8—115.6)

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 the improvement, in a method of reducing the shrinking tendencies of a wool-containing textile material with an aminoplast adapted to effect this result, which comprises treating the said textile material with an alkaline solution, more particularly an aqueous solution or an organic solvent (e. g., an alcoholic) solution of a peroxy compound (e. g., hydrogen peroxide, an organic or inorganic peroxide, a perborate, a persulfate, etc.) prior to treatment with the aminoplast.

Many different methods and agents have been used or suggested for use in the treatment of fabrics or 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, hypochlorites (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 shrinkproofing 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 aminoaldehyde (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 shrink-proofing 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 shrinkproofing 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 shrinkproofing 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 hypoclorite 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, and the aminoplasts therein set forth are among those which are suitable for use in practicing our invention.

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 resistant to felting and shrinking, with the use of a lesser amount of an aminoplast than previously was required, by a particular preconditioning treatment of the textile prior to treatment with the aminoplast, which may be, for example, a methylated or other alkylated methylol melamine. Briefly, the preconditioning treatment comprises treating the textile with an alkaline solution, preferably an aqueous alkaline solution, of a peroxy compound, preferably a water-soluble peroxy compound, prior to treatment with the aminoplast. It was wholly unobvious and unexpected that pretreating the wool-containing textile with an alkaline solution of a peroxy compound would make possible a substantial reduction in the amount of methylated methylol melamine or other aminoplast required to obtain a desired degree of shrinkage resistance together with other advantages described below, especially 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 greater uniformity in the results generally obtained with a given aminoplast in treating a wider variety of textiles composed of or containing a proteinaceous material, specifically wool; and the ability to use, with effective results, other synthetic resins, more particularly aminoplasts other than those heretofore commonly employed in reducing the shrinkage tendencies of a wool-containing textile. Still another advantage accruing from the preconditioning treatment employed in practicing our invention is the ability to render shrink-resistant woolen textiles which heretofore could not be rendered resistant to shrinking to an appreciable or a commercially satisfactory degree by previously known methods.

The reasons why the treatment of a wool-containing textile with an alkaline solution of a peroxy compound prior to treatment with a methylated methylol melamine or other 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 an 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 reconditioning treatment of the undyed wool-containing textile and the final treatment of the textile with an aminoplast.

In carrying our invention into effect we first prepare a 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 solution 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 peroxide; 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 perferably 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 aqueous alkaline solution.

The preferred alkaline solution of the peroxy compound is an alkaline solution of the peroxy compound in water. However, 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 aqueous solution 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, additional basic material may be added to the pretreating bath during the treatment in order to maintain the bath at a desired pH value.

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 about 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 usually 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), with or without further washing with water to remove the excess acid and water-soluble salts as desired or as conditions may require; or, the initial washing with water may be omitted, and merely a dilute acid rinse followed, if desired or deemed necessary, 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. Typical 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 a vat dye.

The final operation in carrying our invention into effect is the treatment of the preconditioned, dyed or undyed, wool-containing textile with a heat-curable aminoplast adapted to render the textile resistant to shrinking when the said aminoplast has been cured under heat to a substantially water-insoluble condition. The preferred heat-curable aminoplasts, more particularly heat-curable or -convertible amidogen-aldehyde (e. g., amidogen-formaldehyde) reaction products are those which are at least partly soluble in water. For example, the heat-curable aminoplast may be a substantially monomeric reaction product or a relatively low molecular weight intermediate reaction product of an aldehyde, specifically formaldehyde, with an amidogen compound containing a plurality of $-NH_2$ groups, for instance urea, melamine, mixtures of urea and melamine, etc. Alkylated (e. g., methylated, ethylated, propylated, butylated, etc.) reaction products of an amidogen compound and an aldehyde, e. g., formaldehyde, may be employed, for instance a methanol- or other alkanol-reacted urea-formaldehyde, melamine-formaldehyde, etc., reaction product. We prefer to use an alkylated, specifically methylated, methylol melamine, more particularly methylated trimethylol melamine. Such products are more fully described in, for example, the aforementioned Johnstone, Jr., et al. Reissue Patent No. 22,566.

The heat-curable aminoplast may be applied to the undyed or dyed wool-containing textile material in various ways. For example, if the heat-curable aminoplast is applied in the form of an aqueous liquid composition (e. g., as a true solution or a colloidal dispersion) containing the same, the dry or substantially dry textile to be treated may be immersed in the aqueous composition 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 heat-curable aminoplast in dissolved or dispersed state is diluted with a suitable solvent (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 or 25% by weight of the composition depending, for example, upon the method of expression of the wet, impregnated textile. Ordinarily, however, the heat-curable aminoplast constitutes from about 3 or 4% to about 10 or 15% by weight of the liquid (e. g., aqueous) finishing composition. The solvent or dispersion medium for the heat-curable aminoplast may be varied depending, for example, upon the characteristics of the aminoplast, but in the case of the preferred aminoplasts it is usually water; or it may be, for instance, a mixture of water and an alcohol, e. g., ethyl alcohol.

In order to speed up the curing of the heat-curable aminoplast or to decrease the heating time various catalysts may be added, if desired, to the finishing composition, e. g., oxalic acid, acetic acid, phosphoric acid, diammonium hydrogen phosphate, diammonium ethyl phosphate, diammonium phthalate, ammonium chloride, ammonium silicofluoride, zinc chloride and others which are known to be catalysts for curing heat-curable aminoplasts, e. g., modified and unmodified melamine-formaldehyde reaction products including the alkylated methylol melamines. The amount of catalyst may be varied as desired or as conditions may require, but ordinarily it is employed in an amount corresponding to from 0.5 to 5% by weight of the heat-curable aminoplast.

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 shrinkproofing 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 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 an aminoplast alone to yield a commercially satisfactory shrinkproofed textile, the pick-up of the liquid finishing composition may be adjusted so that the finished textile contains as much as about 10% or even about 15% or more, by weight of the dry textile, of the cured aminoplast. The amount of the 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 heat-curable aminoplast, nearly the same percentage of cured aminoplast as the percentage of heat-curable 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 160° F. to about 400° F. are employed. The impregnated textile may be air dried or it may be dried under heat, e. g., at a temperature of the order of about 160° F. to 212° F. to remove all or most of the water, after which the dried material is heated at a temperature within the range of about 250° F. to about 400° F. to convert or cure the heat-curable aminoplast to a substantially water-insoluble condition.

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 tenter, drying the framed fabric, batching it upon a shell and then allowing it to stand hot for a period sufficient to convert the solid impregnant in situ to a water-insoluble condition. In some cases drying and curing for 2 or 3 hours at 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 250° F. to 400° F. 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 undyed women's wear flannel, 100% all wool, 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 thereof 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 thoroughly 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 10 minutes at 240° F.

B. Another sample of the flannel was impregnated by immersing it for at least 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 A, and then heated for 9 minutes at 290° F. to dry the fabric and to cure the methylated methylol melamine to a substantially water-insoluble condition.

C. Same as described under B with the exception that the flannel was immersed in an aqueous solution containing about 10% of methylated methylol melamine (more particularly methylated trimethylol melamine), 0.31% of diammonium hydrogen phosphate and 0.03% of dioctyl sodium sulfosuccinate.

D. A sample of the flannel was preconditioned as described under A, followed by treatment 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 or more 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 (control sample which had been similarly washed), is shown in Table I. Other shrinkage values given in other examples were determined in a similar manner.

Table I

WARP SHRINKAGE IN PER CENT

	No. of Machine Washings		
	10	20	30
Untreated flannel	22.0	------	-----
Treated flannel of A	13.9	------	-----
Treated flannel of B	7.2	10.0	11.9
Treated flannel of C	5.3	5.0	6.1
Treated flannel of D	4.4	6.1	6.1

EXAMPLE 2

This example illustrates the results obtained when the women's wear flannel used in Example 1 is treated as therein described but varying the concentration of hydrogen peroxide in the preconditioning bath, the period of time the flannel is immersed in this bath, and the manner of washing after the peroxide pretreatment. The finishing treatment with a 5% aqueous solution of a methylated methylol melamine was the same as described under B of Example 1. The results are shown in Table II, together with warp shrinkage data on the untreated flannel and on samples of flannel which had been treated with 5% and 10% aqueous solutions of methylated methylol melamine as described under B and C of Example 1.

Table II

Test No.	H_2O_2	Time of Treatment	Acetic Acid Rinse After H_2O_2 Treatment	Methylated Methylol Melamine Applied	Warp Shrinkage		
					No. of Machine Washings		
					10	20	30
	Per cent	*Hours*		*Per cent*	*Per cent*	*Per cent*	*Per cent*
1	0	----------	----------	0	22.0	--------	--------
2	0.6	½	No	0	16.9	--------	--------
3	0	----------	----------	5	7.2	10.0	11.9
4	0	----------	----------	10	5.3	5.0	6.1
5	0.6	3	No	5	6.7	6.9	7.8
6	0.6	1½	No	5	5.0	5.8	6.4
7	0.6	½	No	5	4.2	5.0	6.1
8	0.6	½	Yes	5	4.4	4.7	5.6
9	0.6	¼	Yes	5	5.8	6.7	6.9
10	0.15	½	Yes	5	5.8	6.7	6.4
11	0.3	¼	Yes	5	6.9	8.0	8.3
12	0.3	½	Yes	5	4.2	5.0	5.8
13	1.2	½	No	5	6.7	6.1	--------
14	2.4	½	No	5	6.9	6.9	--------

In Table II the word "No" under the fourth column heading means that the peroxide pretreated cloth was not given an acetic acid rinse but was merely rinsed in cold water and dried as described under A of Example 1. The word "Yes" under this same column heading means that the peroxide pretreated cloth was first rinsed in water, then in a dilute acetic acid solution containing about 5% by weight of acetic acid, and finally with water to remove the acid. The same meanings apply to these words where they appear in other tables herein under the column heading "Acetic acid rinse after H_2O_2 treatment."

EXAMPLE 3

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 to render them adequately resistant to shrinking than the flannel used in Examples 1 and 2. The pretreating bath was an aqueous alkaline solution containing 0.6% by weight thereof of hydrogen peroxide and rendered alkaline to a pH of about 10 with sodium silicate.

The other conditions of treatment with the peroxide preconditioning solution and with the methylated methylol melamine were the same as described under Examples 1 and 2. The results are shown in Table III, together with warp shrinkage data on the untreated flannel and on samples of flannel which had been treated with 5% and 10% aqueous solutions of methylated methylol melamine as described under B and C of Example 1, and also with an aqueous solution containing 15% by weight thereof of methylated methylol melamine, 0.465% of diammonium hydrogen phosphate and 0.03% of dioctyl sodium sulfosuccinate.

Table III

H_2O_2	Time Treatment	Acetic Acid Rinse After H_2O_2 Treatment	Methylated Methylol Melamine Applied	Warp Shrinkage			Total Tensile Strength (Warp Plus Filling)
				No. of Machine Washings			
				10	20	30	
Per cent	*Hours*		*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Pounds*
0			0	44.5	49.0		69
0			5	31.0			
0			10	10.8	20.8		
0.6	½	No	5	5.8	7.2	9.2	72
0.6	½	Yes	5	5.6	6.7	7.5	75
0.6	3	No	5	5.3	6.7	7.2	
0.6	3	Yes	5	4.2	5.6	5.6	

The alkaline hydrogen peroxide pretreatment stripped the color from the yellow wool, 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 5% methylated methylol melamine solution.

EXAMPLE 4

Same as Example 3 with the exception that the textile material was a 100% all wool, 8-ounce flannel which had been carbonized in a particular manner. The flannel was an undyed wool, but like the flannel used in Example 3 it is not readily amendable to treatment with a methylated methylol melamine alone to render it shrinkage-resistant. Because of the residual sulfuric acid in this carbonized flannel, about three times the normal amount of sodium silicate was required to provide and keep the pretreating bath at a pH of about 10 during the peroxide pretreatment. The results are shown in Table IV, together with warp shrinkage data on the untreated flannel and on a sample of the flannel which had been treated with a 10% aqueous solution of methylated methylol melamine as described under C of Example 1.

Table IV

H_2O_2	Time of Treatment	Acetic Acid Rinse After H_2O_2 Treatment	Methylated Methylol Melamine Applied	Warp Shrinkage			Total Tensile Strength (Warp Plus Filling)
				No. of Machine Washings			
				10	20	30	
Per cent	*Hours*		*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Pounds*
0			0	30.8	45.0		76
0			10	9.4	14.7		
0.6	3	No	5	1.7	2.5	3.1	
0.6	½	Yes	5	3.6	4.4	5.0	79
0.6	3	Yes	5	6.7	7.5	8.0	75

EXAMPLE 5

Same as Examples 3 and 4 with the exception that another undyed, 100% all wool, 8-ounce flannel goods, which is not readily amenable to treatment with a methylated methylol melamine alone to render it shrinkage-resistant, was employed. The results are shown in Table V. As indicated in this table, one of the samples was top-chrome dyed after the peroxide pretreatment and before the finishing treatment with the 5% aqueous methylated methylol melamine solution.

Table V

H_2O_2	Time of Treatment	Acetic Acid Rinse After H_2O_2 Treatment	Methylated Methylol Melamine Applied	Warp Shrinkage			Total Tensile Strength (Warp Plus Filling)
				No. of Machine Washings			
				10	20	30	
Per cent	*Hours*		*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Pounds*
0			0	43.9			74
0			10	7.2	11.4	12.8	
0.6	3	No	5	5.3	6.1	6.4	
0.6	3	No	5	4.4	4.7	5.0	
0.6	½	No	5	5.3	5.3	5.3	76
0.6	½	Yes	5	3.9	4.4	4.7	75
0.6	[1]3	No	5	3.3	4.2		

[1] NOTE: After the peroxide pretreatment the sample was top-chrome dyed prior to treatment with the aqueous methylated methylol melamine. The wool dyed satisfactorily after the peroxide pretreatment without detracting from the improved shrinkage resistance obtained upon subsequent treatment of the dyed wool with the aqueous methylated methylol melamine.

EXAMPLE 6

In this example 100% all wool, women's wear flannel weighing 8 ounces per square yard was treated as described briefly below, the details of the steps being the same as described in prior examples.

A. The wool was immersed for 30 minutes in an aqueous solution of sodium silicate in a concentration such as to give the solution a pH of about 10. The solution was maintained at 125° F. during the treating period. The cloth was removed from the treating bath, rinsed in cold water and dried.

B. Same as A with the exception that the pretreating solution was an aqueous solution containing only 0.6% by weight thereof of hydrogen peroxide.

C. Same as A with the exception that the pretreating solution contained 0.6% by weight thereof of hydrogen peroxide and was rendered alkaline with sodium silicate to a pH of about 10.

D. The flannel, without any preconditioning treatment, was treated with a 5% aqueous solution of methylated methylol melamine as described under B of Example 1.

E. The wool was treated as described under A, supra, rinsed first in water, then in a dilute aqueous solution of acetic acid and again in water, dried, and finally treated with a 5% aqueous solution of methylated methylol melamine as described under B of Example 1.

F. Same as E, supra, with the exception that the preconditioning treatment was the same as described under B, supra.

G. Same as E, supra, with the exception that the preconditioning treatment was the same as described under C, supra.

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	18.6
A	16.4
B	18.0
C	13.9
D	6.7
E	8.9
F	8.3
G	3.9

EXAMPLE 7

The same kind of flannel was treated as that employed in Example 6. The treatments were as follows:

A. The flannel, without any preconditioning treatment, was treated with an aqueous solution containing 10% of trimethylol melamine, 0.31% of diammonium hydrogen phosphate and 0.03% of dioctyl sodium sulfosuccinate.

B. The woolen goods was preconditioned by treatment as described under A of Example 6, rinsed first in water, then in a dilute aqueous solution of acetic acid and again in water, dried, and finally treated as described under A of this example.

C. Same as B of this example with the exception that the preconditioning treatment was the same as described under B of Example 6.

D. Same as B of this example with the exception that the preconditioning treatment was the same as described under C of Example 6.

The treated flannels and a control sample were washed as described under Example 6. The shrinkage data on the treated and washed samples are shown below:

Treatment	Warp Shrinkage in Per Cent
None	18.6
A	8.0
B	8.0
C	7.8
D	4.2

EXAMPLE 8

The same kind of flannel was treated as that employed in Example 6. The treatments were as follows:

A. The flannel, without any preconditioning treatment, was treated with an aqueous solution containing 8% of methylated dimethylol urea, about 0.25% of diammonium hydrogen phosphate and 0.03% of dioctyl sodium sulfosuccinate.

B. The woolen goods was preconditioned as described under A of Example 6, rinsed first in water, then in a dilute aqueous solution of acetic acid and again in water, dried, and finally treated as described under A of this example.

C. Same as B of this example with the exception that the preconditioning treatment was the same as described under B of Example 6.

D. Same as B of this example with the exception that the preconditioning treatment was the same as described under C of Example 6.

The treated flannels and a control sample were washed as described under Example 6. The shrinkage data on the treated and washed samples are shown below:

Treatment	Warp Shrinkage in Per Cent
None	18.6
A	17.2
B	17.8
C	20.6
D	8.3

EXAMPLE 9

The textile material employed in this example was a Neolan dyed, 100% all wool flannel such as was used in Example 3. The treatments were as follows:

A. Same as A of Example 6.

B. Same as C of Example 6.

C. Same as G of Example 6.

D. Same as D of Example 8.

E. Same as D of Example 7.

The alkaline hydrogen peroxide pretreatment used in B, C, D and E stripped the color from the yellow wool, but did not detract from the ability of the peroxide to precondition the cloth and thereby obtain improved shrinkage resistance upon subsequent treatment with the heat-curable aminoplast.

The treated flannels and a control sample were washed as described under Example 6. The shrinkage data on the treated and washed samples are shown below:

Treatment	Warp Shrinkage in Per Cent
None	44.0
A	36.1
B	20.6
C	4.2
D	11.4
E	3.3

EXAMPLE 10

In this example a different peroxy compound, viz., sodium perborate, was employed. The fabric treated was a women's wear flannel such as was used in Example 1. The treatments were as follows:

A. Same as B of Example 1, that is, the flannel was not preconditioned, and the textile-finishing composition comprised a 5% aqueous solution of methylated methylol melamine.

B. Same as C of Example 1. The flannel was not pretreated and the finishing composition comprised a 10% aqueous solution of methylated methylol melamine.

C. Same as A of Example 1 with the exception that, instead of the preconditioning bath containing 0.6% by weight of hydrogen peroxide, it contained 1% by weight of sodium perborate.

D. The flannel was preconditioned by immersing it in an aqueous alkaline solution containing 1% by weight of sodium perborate, which bath differed from the solution of C of this example in that no sodium silicate was added. (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. After rinsing with water and drying as described under A of Example 1, the preconditioned flannel was treated with a solution containing 5% by weight of methylated methylol melamine as described under B of Example 1.

The treated flannels and a control sample were washed as described under Example 6. The shrinkage data on the treated and washed samples are shown below:

Treatment	Warp Shrinkage in Per Cent
None	22.0
A	8.9
B	5.3
C	14.2
D	4.4

EXAMPLE 11

The woolen material which was treated in this example was the same as that used in Examples 6, 7 and 8. The treatments were as follows:

A. Same as D of Example 1 with the exception that the hydrogen peroxide preconditioning bath, which had been rendered alkaline to a pH of about 10 with sodium silicate, was maintained at about 150° F. during the period the flannel was immersed in the bath. The pretreating solution contained 0.6% by weight of hydrogen peroxide. The final treatment was with a solution containing 5% by weight of aqueous methylated methylol melamine as described more fully under B of Example 1.

B. Same as A of this example with the exception that the perconditioning solution contained 1.2% by weight of hydrogen peroxide.

C. Same as A of this example with the exception that the pretreating solution contained 2.4% by weight of hydrogen peroxide.

The treated flannels were washed as described under Example 6. The shrinkage data on the treated and washed samples are shown below:

Treatment	Warp Shrinkage in Per Cent
A	5.8
B	5.6
C	5.6

EXAMPLE 12

The woolen fabric which was treated in this example was a 100% all wool, women's wear flannel weighing 8 ounces per square yard. The treatments were as follows:

A. Same as D of Example 1 with the exception that the hydrogen peroxide preconditioning solution was rendered alkaline to a pH of about 10 with sodium carbonate instead of with sodium silicate as in Example 1–D.

B. Same as D of Example 1 with the exception that the preconditioning bath was rendered alkaline to a pH of about 10 with guanidine carbonate.

C. Same as D of Example 1 with the exception that the hydrogen peroxide pretreating solution was brought to a pH of about 9 with triethanolamine.

D. Same as D of Example 1 with the exception that the preconditioning bath was rendered alkaline to a pH of about 8 with sodium acetate.

E. Same as D of Example 1 with the exception that the peroxide preconditioning solution was made alkaline to a pH of about 10 with sodium hydroxide.

F. Same as D of Example 1 with the exception that the peroxide pretreating solution was brought to a pH of about 8 with soap.

The treated goods and a control sample were washed as described under Example 6. The shrinkage data on the treated and washed samples are shown below:

Treatment	Warp Shrinkage in Per Cent
None	26.0
A	4.2
B	3.9
C	6.4
D	9.2
E	8.6
F	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 solution containing 5% of methylated dimethylol urea, 0.155% of diammonium hydrogen phosphate and 0.03% of dioctyl sodium sulfosuccinate.

B. Same as A with the exception that the flannel was treated with an aqueous solution containing 10% of methylated dimethylol urea, 0.31% of diammonium hydrogen phosphate and 0.03% of dioctyl sodium sulfosuccinate.

C. Same as A with the exception that the flannel was preconditioned by treatment with an aqueous alkaline solution containing 0.6% by weight of hydrogen peroxide as described under A of Example 1.

D. Same as B with the exception that the flannel was pretreated with an aqueous alkaline hydrogen peroxide solution as described under A of Example 1.

The shrinkage data on the treated samples, after 5 washing cycles of 10 minutes each followed by drying and, also, after washing for an additional hour followed by drying, 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	15.0	
B	11.1	
C	5.6	5.8
D	5.3	5.3

EXAMPLE 14

Same as Example 13 with the exception that instead of the aminoplast employed in B and D of that example there was used a 10% aqueous solution of a substantially unpolymerized reaction product of formaldehyde and urea in the ratio of about 1.33 moles of the former to 1 mole of the latter (B and D of this example); and instead of the aminoplast employed in C of Example 13 there was used a 5% aqueous solution of the same aminoplast just mentioned (C of this example). In each case the solution also contained 0.03% of dioctyl sodium sulfosuccinate, and diammonium hydrogen phosphate in an amount corresponding to about 3.1% by weight of the aminoplast in the solution.

The shrinkage data on the treated samples after being washed are shown below:

Treatment	Warp Shrinkage in Per Cent, After 5 Washing Cycles
B	16.7
C	12.2
D	12.5

EXAMPLE 15

Same as Example 13 with the exception that instead of the aminoplast employed in B and D of that example there was used a 10% aqueous solution of a partial reaction product of urea and formaldehyde in the ratio of 1 mole of the former to from 2.0 to 2.1 moles of the latter; and instead of the aminoplast employed in C of Example 13 there was used a 5% aqueous solution of the aminoplast just mentioned (C of this example). In each case the solution also contained 0.03% of dioctyl sodium sulfosuccinate and diammonium hydrogen phosphate in an amount corresponding to about 3.1% by weight of the aminoplast in the solution.

The shrinkage data on the treated samples after being washed are shown below:

Treatment	Warp Shrinkage in Per Cent	
	After 5 Washing Cycles of 10 Minutes Each	After 1 Hour of Additional Washing
B	10.5	
C	5.6	6.1
D	4.4	4.7

EXAMPLE 16

Same as D of Example 1 with the exception that the flannel was immersed in an aqueous solution containing 2½% by weight of methylated methylol melamine. The warp shrinkage in per cent of the treated flannel, after 5 washing cycles of 10 minutes each followed by 1 hour's washing was 4.2.

EXAMPLE 17

Same as D of Example 15 with the exception that, instead of drying and curing the impregnated flannel by heating for 9 minutes at 290° F., the period and temperature of heating were, in one case (A), 9 minutes at 225° F. and, in another case (B), 15 minutes at 290° F. The shrinkage data on the treated samples after being washed are shown below:

Treatment	Warp Shrinkage in Per Cent[1]
A	6.1
B	3.3

[1] After 5 Washing Cycles of 10 Minutes Each Plus One Hour's Additional Washing.

EXAMPLE 18

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 was a 100% all wool, women's wear flannel such as was used in Example 1. The treatments were as follows:

A. Same as D of Example 1 with the exception that the woolen fabric was immersed in the alkaline hydrogen peroxide pretreating bath at a temperature of 100° F. for 8 hours instead of at 125° F. for 30 minutes as in 1–D. The textile-finishing composition contained about 5% of methylated trimethylol melamine and the same kind and amount of curing catalyst and wetting agent as in 1–D.

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

The treated flannels and a control sample were washed as described under Example 6. 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	4.4

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

It will be understood, of course, by those skilled in the art that our invention is not limited to the particular preconditioning compositions, heat-curable aminoplasts, curing catalyst 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.

In the copending application of Linton A. Fluck, Jr., John E. Lynn and Eleanor L. McPhee, Serial No. 43,964, filed concurrently herewith, is disclosed and claimed a process of reducing the shrinking tendencies of a wool-containing textile material which comprises preconditioning the textile material by first treating it with an alkaline solution of a peroxy compound as herein described, impregnating the preconditioned textile with an impregnant comprising a colloidal solution of a curable cationic aminoplast (e. g., a curable, cationic product of reaction of ingredients comprising melamine and formaldehyde), and advancing the cure of the said aminoplast in situ, the amount of the cured aminoplast with which the said textile is impregnated being sufficient to render it resistant to shrinking.

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 curable, non-cationic aminoplast, and advancing the cure of the said aminoplast in situ, the amount of cured aminoplast in the said textile material being sufficient to render it resistant to shrinking.

2. A process as in claim 1 wherein the alkaline solution of the peroxy compound has a pH of at least 8.

3. A process as in claim 1 wherein the alkaline solution is an aqueous alkaline solution and the peroxy compound is a water-soluble peroxide.

4. A process of reducing the shrinking tendencies of a woolen fabric material which comprises preconditioning the said fabric material by first treating it with an aqueous alkaline solution of hydrogen peroxide and thereafter removing the excess alkaline material, impregnating the preconditioned fabric with an impregnant comprising a curable, non-cationic aminoplast which is a reaction product of ingredients comprising melamine and formaldehyde, and advancing the cure of the said aminoplast in situ, the amount of cured aminoplast in the said fabric material being sufficient to render it resistant to shrinking.

5. A process of reducing the shrinking tendencies of a dyed, wool-containing tetxile material which comprises preconditioning the undyed textile material by first treating it with an aqueous alkaline solution of a water-soluble peroxide, dyeing the preconditioned textile material, impregnating the dyed textile material with a curable, non-cationic aminoplast, and advancing the cure of the said aminoplast in situ to its ultimate form, the cured aminoplast being substantially water-insoluble, and the amount of cured aminoplast in the said textile material being from about 1% to about 15% by weight of the dry, untreated textile material.

6. A process as in claim 5 wherein the aqueous alkaline solution has a pH of between 8 and 12.

7. A process of reducing the shrinking tendencies of a dyed, wool-containing fabric material which comprises immersing the undyed fabric material in an aqueous alkaline solution of hydrogen peroxide and thereafter removing the excess alkaline material thereby to precondition the undyed fabric, dyeing the preconditioned fabric, impregnating the dyed fabric with an aqueous composition comprising a curable, non-cationic aminoplast which is a reaction product of ingredients comprising melamine and formaldehyde, drying the wet, impregnated fabric material and polymerizing the said curable aminoplast contained in the impregnated fabric to its ultimate degree of polymerization, said drying and polymerization being effected at a temperature not exceeding 400° F., and the amount of polymerized aminoplast in the said fabric being from 1 to 15% by weight of the dry, untreated fabric.

8. A process as in claim 7 wherein the hydrogen peroxide is employed in a concentration of at least 0.05% by weight of the aqueous alkaline solution.

9. A process as in claim 7 wherein the aqueous solution is rendered alkaline with sodium silicate to a pH of between 9 and 11.

10. A process of reducing the shrinking tendencies of a dyed, woolen fabric material which comprises immersing the woolen fabric material, prior to dyeing, for a period of at least 15 minutes in an aqueous solution rendered alkaline with sodium silicate to a pH of between 9 and 11 and containing hydrogen peroxide in an amount corresponding to from 0.05 to 5% by weight of the said solution, said solution being maintained at a temperature within the range of 70° F. to 160° F., thereafter removing the excess sodium silicate from the thusly treated fabric material thereby to precondition the undyed fabric, dyeing the preconditioned fabric, impregnating the dyed fabric with an aqueous solution of a non-cationic methylated methylol melamine, drying the wet, impregnated fabric and polymerizing the methylated methylol melamine contained in the impregnated fabric to its ultimate degree of polymerization, said drying and polymerization being effected at a temperature within the range of about 65° F. to about 300° F., and the amount of polymerized methylated methylol melamine contained in the said fabric being from about 1% to about 15% by weight of the dry, untreated fabric.

11. The method of producing a dyed wool-containing textile material which is resistant to shrinking, said method including, as essential steps, treating the undyed wool-containing textile material with an aqueous solution rendered alkaline to a pH of between 8 and 12 and containing hydrogen peroxide in an amount corresponding to from 0.1 to 3% by weight of the said solution thereby to precondition the said textile material, dyeing the preconditioned textile material, impregnating the dyed preconditioned textile material with a curable, non-cationic product of partial reaction of ingredients com-

prising melamine and formaldehyde, and thereafter subjecting the impregnated wool-containing textile material to an elevated temperature sufficient to cure the said reaction product contained therein to a substantially water-insoluble condition, the amount of the cured reaction product with which the said textile material is impregnated being sufficient to render it resistant to shrinking and constituting from about 1% to about 15% by weight of the dry untreated textile material.

12. The method of producing a dyed woolen fabric material which is resistant to shrinking, said method comprising treating the undyed woolen fabric material with an aqueous solution rendered alkaline with sodium silicate to a pH of between 9 and 11 and containing hydrogen peroxide in an amount corresponding to from 0.2 to 2.5% by weight of the said solution thereby to precondition the said fabric material, removing the excess sodium silicate from the thusly treated material, dyeing the resulting material, impregnating the dyed material with an aqueous solution of a non-cationic methylated methylol melamine, drying the wet impregnated material, and thereafter heating the dried material at a temperature within the range of 250° F. to 400° F. to cure the methylated methylol melamine contained therein to a substantially water-insoluble condition, the amount of cured methylated methylol melamine with which the said fabric material is impregnated being sufficient to render it resistant to shrinking, said cured methylated methylol melamine constituting from about 2% to about 10% by weight of the dry untreated fabric material and being substantially less than that required to produce the same degree of shrinkage resistance in the same woolen fabric material with the same methylated methylol melamine in the absence of the aforesaid hydrogen peroxide preconditioning treatment.

13. 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 solution of a curable, non-cationic methylated methylol melamine, said solution containing from about 3% to about 15% by weight of the said methylated methylol melamine and a curing catalyst therefor in an amount corresponding to from 0.5% to 5% by weight of the methylated methylol melamine; adjusting the pick-up of the said aqueous solution by the said preconditioned textile so that the amount of cured methylated methylol melamine in the finished textile is from about 2% to about 10% by weight of the dry untreated textile and 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 heating the dried textile at a temperature within the range of 250° F. to 400° F. to cure the methylated methylol melamine contained therein to a substantially water-insoluble condition.

14. The method of producing a wool-containing textile material which is resistant to shrinking, said method including, as essential steps, first treating the said textile material with an aqueous solution rendered alkaline to a pH of between 8 and 12 and containing hydrogen peroxide in an amount corresponding to from 0.1 to 3% by weight of the said solution thereby to precondition the said textile material, impregnating the preconditioned textile material with a curable, non-cationic aminoplast, and thereafter subjecting the thusly impregnated wool-containing textile material to an elevated temperature sufficient to cure the said aminoplast contained therein to a substantially water-insoluble condition, the amount of cured aminoplast with which the said textile material is impregnated constituting from about 1% to about 15% by weight of the dry, untreated textile material.

15. A method as in claim 14 wherein the curable, non-cationic aminoplast is a curable, non-cationic product of partial reaction of ingredients comprising melamine and formaldehyde.

LINTON A. FLUCK, Jr.
JOHN E. LYNN.
EDWARD A. SCHUMAN.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,389,274	Schweitzer	Aug. 30, 1921
2,070,210	Mason	Feb. 9, 1937
2,107,297	Kauffmann	Feb. 8, 1938
2,238,949	Schlack	Apr. 22, 1941
2,329,622	Johnstone	Sept. 14, 1943
2,329,651	Powers	Sept. 14, 1943
2,395,724	Cowley	Feb. 26, 1946
2,395,791	Pfeffer	Feb. 26, 1946
2,467,233	Rust	Apr. 12, 1949

FOREIGN PATENTS

Number	Country	Date
408,985	Great Britain	Apr. 20, 1934
553,923	Great Britain	1943

Certificate of Correction

Patent No. 2,539,365 January 23, 1951

LINTON A. FLUCK, Jr., ET AL.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows:

Column 2, line 27, for "hypoclorite" read *hypochlorite*; column 3, line 48, for "reconditioning" read *preconditioning*; column 4, line 20, for "perferably" read *preferably*; column 7, line 30, for "silocofluoride" read *silicofluoride*; column 12, line 2, for "amendable" read *amenable*; column 17, line 48, for "Varp" read *Warp*; column 19, line 70, for "tetxile" read *textile*;

and that the said Letters Patent should be read as corrected above, so that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 10th day of April, A. D. 1951.

[SEAL]

THOMAS F. MURPHY,
Assistant Commissioner of Patents.