

[54] PERMANENT PRESS MODIFICATION OF CELLULOSIC FABRICS

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[58] Field of Search 260/256.4 C, 256.4 F; 8/116.3, 115.7; 38/144

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[57] **ABSTRACT**

The compound, 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine, is reacted with between about 2.80 molar proportions and about 5.0 molar proportions of formaldehyde in aqueous medium to produce an aminoplast material. When applied to a fabric and cured, the aminoplast material forms a finish which imparts permanent press properties to the fabric.

5 Claims, No Drawings

PERMANENT PRESS MODIFICATION OF CELLULOSIC FABRICS

BACKGROUND OF THE INVENTION

This invention relates to a novel aminoplast material. The invention also relates to a process for producing the aminoplast material and to the method for using same to impart "permanent press" properties to fabrics, particularly cellulosic fabrics.

The term "permanent press resins" is understood to mean substances which, when applied to fabrics, impart to the fabrics properties of "wrinkle resistance" and "durable press". The property of wrinkle resistance (which is synonymous with terms such as "wrinkle-recovery", "crease resistance", and the like) refers to the ability of a fabric to resist wrinkling and to retain smoothness of shape and hand upon repeated wear and laundering. The property of durable press (which is synonymous with terms such as "crease retention" and the like) is understood to mean the ability of a treated fabric to drip-dry without loss of crease and to be worn without ironing, although fastidious wearers may insist on some "touch-up" ironing.

Various methods are known for imparting permanent-press properties to fabrics, particularly fabrics derived from cellulosic materials (e.g., "regenerated" cellulosic materials such as rayon and "natural" cellulosic materials such as cotton and cotton/polyester blends and the like). However, these prior art techniques suffer a number of disadvantages. Thus, for example, some processes heretofore employed to impart permanent-press properties to cellulosic fabrics result in finished products which when bleached with the usual hypochlorite bleaching solutions, retain an undesirable amount of chlorine, which is released as hydrochloric acid upon subsequent exposure to heat, e.g., ironing, thereby causing damage to the fabrics. Moreover, it is generally recognized in the fabric finishing art that fabrics treated with heretofore known permanent press resins tend to be discolored and will experience further substantial and unsightly discoloration on scorching. Accordingly, the overall performance of heretofore known textile finishing agents leaves much to be desired. Thus, for permanent press white cottons, textile finishing agents should impart to the substrate fabric a finish which does not suffer nor otherwise cause acid damage to the substrate fabric as a result of hypochlorite bleaching, and should be durable to alkaline conditions normally encountered in laundering.

Therefore, it is an object of the present invention to provide a novel composition of matter which, when applied to a fabric, particularly a cellulosic fabric, imparts a colorless, permanent press finish thereto.

Another object is to provide a process for producing a novel composition of matter which, when applied to a fabric, and particularly to a cellulosic fabric, imparts a colorless, permanent press finish thereto.

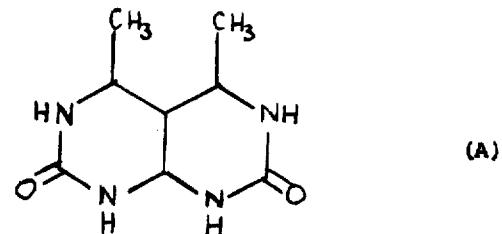
Another object is to provide a permanent press fabric finish.

Yet another object is to provide a process for applying to a fabric, and particularly to a cellulosic fabric, a permanent press finish.

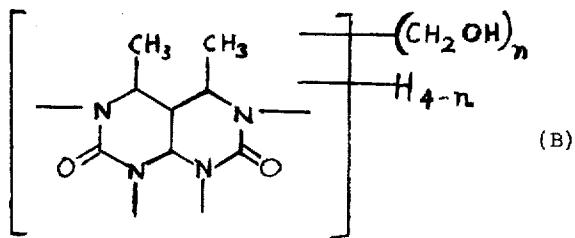
These and other objects of the present invention, as well as a fuller understanding of the advantages thereof can be had by reference to the following detailed description and claims.

SUMMARY OF THE INVENTION

The above objects are achieved according to the present invention by the discovery of a novel composition of matter which, when applied to a fabric, particularly a cellulosic textile fabric, forms a fabric finish with excellent permanent press properties. More particularly, this novel composition of matter is produced according to the present invention by contacting 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine, (A) which has the following formula:



with a specific amount of formaldehyde under carefully controlled reaction conditions. The product resulting from the reaction of compound (A), above, with formaldehyde according to the present invention is a novel aminoplast material, (B), comprising mono-, di-, tri-, and tetramethylolated 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine. The various compounds constituting this aminoplast material are collectively represented by following structural formula:



wherein n represents an integer from 1 to 4, inclusive.

The compound 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine, is reported in 92 *Monatshefte fuer Chemie* 31 (1961) as a crystalline solid melting at 277°C. This material can be prepared according to the aforementioned reference by the acid-catalysed reaction between acetaldehyde and urea in aqueous menstruum. The ratio of the concentration of acetaldehyde to that of urea in the reaction system is at least about 1.5, and preferably between about 1.8 and about 2.2. The reaction is desirably conducted at atmospheric pressure and at an elevated temperature, preferably at a temperature of between about 50°C. and about 100°C., with the atmospheric reflux temperature of the reaction system being especially preferred.

It is a feature of the present invention that the 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine must be employed in a high state of purity, i.e., in crystalline form. In addition, it is especially important that this material be free of color bodies which become noticeable when the aminoplast (B) is applied to a fabric, or during the lifetime of the treated fabric.

The compound 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine suitable for use

in the present invention is conveniently and preferably prepared according to the following procedure, it being understood, of course, that other methods of synthesis can be used without departing from the spirit of the invention. Thus 5285 parts by weight (120 moles) of acetaldehyde are charged to a suitable conventional reaction vessel and cooled to below 20°C. Then a solution of 3600 parts by weight (60 moles) of urea and 645 parts by weight of reagent grade sulfuric acid (98 percent) in 4275 parts by weight of water are added dropwise with stirring to the acetaldehyde. During the addition, a suitable cooling means (e.g., an ice-bath) is applied to the reaction vessel, and the rate of addition is adjusted, if necessary, in order to maintain the reaction system at a temperature of about 20°C. When the addition is complete, the reaction mixture is heated to atmospheric reflux temperature and maintained thereat for 1 hour. Thereafter, the reaction mixture is cooled to ambient temperature, diluted with water, and filtered to isolate the crystalline product, (A). The product is washed with water, then with methanol, and finally recrystallized from water. After drying the recrystallized product at 70°C., it weighs 1467 parts (25 percent of the theoretical yield based on urea) and melts at 275°-280°C. Elemental analysis of the product corresponds to the formula $C_8H_{14}N_4O_2$.

The reaction of 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine, (B), with formaldehyde to produce the novel aminoplast material of the present invention is accomplished by contacting (A) with between about 2.80 and about 5.0 molar equivalents of formaldehyde in a hydroxylic medium. The hydroxylic solvent used should be capable of dissolving the reactants and also capable of being separated from the aminoplast material formed in the reaction by evaporative means. The preferred solvent is water. The concentrations of the reactants in the hydroxylic medium are not critical provided dissolution of the reactants can take place. In applying the novel aminoplast material of the present invention to a fabric, it is advantageous to do so by simply immersing the substrate fabric into an aqueous solution ("pad bath") of the aminoplast material, said solution being simply the aqueous reaction mixture in which the aminoplast material is formed, modified, of course, by the addition thereto of a suitable curing catalyst or catalyst system of the type described hereinbelow. Accordingly, the concentrations of reactants are preferably those which result in a concentration of aminoplast material which in turn results in the desired amount of resin "pick-up" experienced by the fabric within the pad bath.

In an especially preferred mode of the present process the reaction is conducted in formalin, i.e., an aqueous solution of formaldehyde in which the concentration of the latter is between about 35 and about 40 percent. Alternatively, paraformaldehyde can be used in lieu of or in addition to formalin as the formaldehyde source. The amount of formaldehyde used can range between 2.80 and 5.0 molar proportions, and preferably between about 2.90 and about 3.05 molar proportions per molar proportion of 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine. The use of formaldehyde in amounts such that the molar ratio of formaldehyde to (A) is less than about 2.80:1 is undesirable because the product aminoplast would in such case be contaminated with substantial quantities of materials which tend to inhibit the performance of the

aminoplast material as a permanent press resin. On the other hand, the use of a formaldehyde to 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine molar ratio in excess of about 3.05:1 is also less desirable, and the use of ratio in excess of about 5:1 is particularly undesirable. The reason for this is believed to be that when a quantity of formaldehyde is present which substantially exceeds that which is stoichiometrically required for the formation of trimethylol 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine, such excess tends to contaminate the aminoplast material with formaldehyde and with by-products derived therefrom during the course of evaporative distillation to remove the excess formaldehyde or during curing operations, these being processes which generally require elevated temperatures.

The reaction between 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine and formaldehyde according to the present invention is catalysed by acid or base. In aqueous media, the reaction can be conducted at a pH of less than or greater than 7. Desirably, the reaction is conducted under basic conditions. Preferably, the reaction is conducted at a pH of greater than about 8 and less than about 12, with a pH of between about 9 and about 10 being especially preferred.

The temperature and pressure at which the 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine and formaldehyde are reacted are not critical. Desirably, the reaction is conducted at an elevated temperature and atmospheric pressure, with a temperature corresponding to the atmospheric reflux temperature of the reaction menstruum being preferred. The time required for completion of the reaction is generally less than 1 hour. The exact time required depends upon the reaction temperature, concentration of the catalyst (i.e., the pH) and reactants. A conventional titration method for determining when a particular reaction system has reached equilibrium is referred to in concentration with Example I hereinbelow.

The aminoplast material (B) of the present invention is water-soluble, and is conveniently applied to a fabric in the form of an aqueous solution. Accordingly, the reaction mixture, and preferably the aqueous reaction mixture in which the aminoplast material is formed, can be used directly as the pad bath solution into which the fabric to be treated is immersed or "dip treated". The fabric is then wrung out by conventional means (e.g., between a pair of rollers) to remove excess liquid, and subsequently cured.

The curing of the aminoplast resin on the substrate fabric is performed according to the present invention under conditions of elevated temperature and acid catalysis. A temperature of between about 275°F. and about 425°F. is generally effective in curing the instant aminoplast material to form a permanent press finish on the treated fabric. Preferably, a curing temperature of between about 300°F. and about 350°F. is employed. The time required for curing depends upon the curing temperature and catalyst concentration. Desirably, the curing step is completed between about 5 seconds and about 30 minutes and preferably between about 3 and about 15 minutes. Substances suitable for catalysing the curing process include any conventional acid catalysts heretofore known to be useful in catalysing the curing of conventional aminoplast materials. Such catalysts are employed in conventional amounts, e.g., at concentrations of between about 1 percent and about

50 percent by weight based on the weight of aminoplast materials and includes as a general category those water-soluble inorganic salts which behave as latent acid catalysts, e.g., ammonium chloride, magnesium chloride, zinc nitrate, and the like.

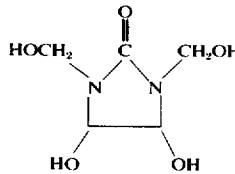
According to a preferred mode of carrying out the fabric treatment process of the present invention, the aqueous reaction mixture containing the novel aminoplast material (B) dissolved therein is cooled to ambient temperature, brought to a pH of about 7.0, and filtered to remove any insolubles which may be present. Then, a conventional amount of a curing catalyst is dissolved in the filtrate, and the fabric to be treated is immersed therein. The amount of resin "picked up" by the substrate fabric is determined in large measure by the concentration of the aminoplast material in the aqueous pad-bath solution. Generally, the concentration of the aminoplast material in the pad bath solution (which can be determined gravimetrically) ranges between about 2 percent or less and about 65 percent by weight or more for cellulosic fabrics. Preferably, a pad-bath concentration of between about 5 percent and about 45 percent is used, with a concentration of between 10 percent and about 25 percent being especially preferred. The particular desired concentration of aminoplast material in any given instance is conveniently achieved by appropriate adjustment of the concentrations of reactants (i.e., formaldehyde and 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine) or by the judicious addition of water to an initially relatively highly concentrated solution of aminoplast material.

After saturating the fabric with the pad bath solution, the treated fabric is withdrawn from the bath, wrung between rollers made of an inert material (e.g., metal, ceramic, rubber, and the like), preferably rubber rollers or adjacent, cooperatively functioning stainless steel/rubber rollers, dried and simultaneously or subsequently heat cured at a temperature within the aforementioned range. The heat curing step can, if desired, be conducted by contacting the fabric with heated metal rollers, preferably heated stainless steel rollers.

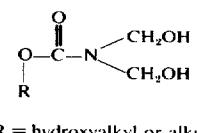
In the present invention, the percent "pickup" of the aminoplast composition is measured as: % Wet Pickup, after immersion in the pad bath solution; % Dry Pickup, after curing; % Dry Pickup, after 1 wash; and % Dry Pickup, after 21 washes.

As will be seen hereinafter, all tests are comparative tests using commercial resins as controls and comparing these resins with the novel composition (B) of the instant discovery. The tests recorded herein, other than resin pickup characteristics, are intended to illustrate the effectiveness of compound (B) with respect to wrinkle recovery, hand, deterioration of fabric soil redeposition, and the like. Obviously, permanent press resins are not attractive if they deleteriously affect fiber strength, if wrinkle recovery is poor, etc.

Fabric, particularly cellulosic fabrics, treated with compound (B) according to the present invention exhibit, as will be seen hereinafter, very desirable and valuable permanent press properties. The examples which follow teach the novel composition of the instant discovery and processes for preparing same. In addition, treatment of fabrics with aminoplast material (B) is fully disclosed, as well as a number of tests comparing the latter's efficacy with that of the following commercially popular resin forming compounds:



10 PERMAFRESH* 113B
(dimethyloldihydroxyethyleneurea)
DMDHEU



15 AEROTEX** 82
(carbamate)

*Trademark for permanent press compound sold by Sun Chemical Co., Wood River Junction, R.I.

**Trademark for permanent press compound sold by American Cyanamid Co., Bound Brook, N.J.

15 The following examples are merely intended to be illustrative of certain of the preferred embodiments within the spirit and scope of the present invention and, therefore, are not to be interpreted too restrictively; parts and percentages given in the examples are by weight, unless otherwise indicated.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

25 The following examples are presented for the purpose of illustrating (but not limiting) the present invention. In the examples, parts and percentages are by weight unless otherwise indicated.

Example I

30 This example illustrates the novel aminoplast material (B) of the present invention and the process for producing same.

To a suitable reaction vessel equipped with conventional agitation means are charged 239 grams (2.94 moles) of 37 percent formalin ("uninhibited") and 15 grams of a 10 percent aqueous solution of sodium carbonate. To the resulting mixture are added, with agitation, 198 grams (1.0 mole) of crystalline 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine. The reaction mixture, which has a pH of between about 9 and about 9.5, is heated to reflux temperature over the course of about 20 minutes and maintained at the reflux temperature for about 5 minutes. The reaction mixture was then cooled to ambient temperature, the pH is adjusted to 7.0, and the reaction mixture filtered to remove insolubles. The clear filtrate contains 2.5 percent free formaldehyde as determined by the "sodium sulfite method" described in Walker, *Formaldehyde*, 3d ed., p. 486 (Reinhold, N.Y., 1964). The concentration of aminoplast material in the aqueous filtrate is 60 percent by weight as determined gravimetrically, i.e., by evaporating an aliquot sample of the solution at 70°C. for 6 hours and weighing the residue.

Example II

55 This example illustrates a variant of the procedure used in Example I to produce the aminoplast material of the present invention.

60 To a suitable reaction vessel equipped with conventional agitation means are charged 2919 grams (36 moles) of 37 percent formalin ("uninhibited") and 135 grams of a 10 percent aqueous solution of sodium carbonate. To the resulting mixture are added, with agitation, 1782 grams (9 moles) of crystalline 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine. The reaction mixture is then heated at reflux temperature for 5 minutes and cooled to ambient temperature. An

additional charge of 135 grams of 10 percent aqueous sodium carbonate and 990 grams (5 moles) of (A) are added and the reaction mixture is again heated to reflux temperature and maintained thereat for 30 minutes. Upon recooling the reaction mixture to ambient temperature, neutralizing the pH, and filtering, a solution of aminoplast material, (B), is obtained which contains 2.0 percent unreacted formaldehyde.

The product 2,7-dioxo-4,5-dimethyl-decahydro-pyrimido-[4,5-d]-pyrimidine/formaldehyde composition (B) of Example II, above, is used to treat plain weave white 50/50 cottonpolyester (PE) cloth, of the type used in shirts or household goods (e.g., sheets and pillowcases). The test procedure is as follows:

Bath:	Compound tested (dry weight basis)	%
	Surfactant (Triton* X-100)	10.0
	Softener (Lubritron** KN)	0.2
	Catalyst-KR (MgCl ₂ solution)***	3.0
	Water	5.0
		81.8
		100.0

Procedure: Standard Pad Bath
70-80% Wet Pickup
Dry at 220°F for 2 minutes
Cure at 325°F for 12 minutes
7-9% Dry Pickup
After wash

Half of the test swatches are laundered
Through 20 cycles.

Tests: Wrinkle Recovery
Stiffness
Tearing Strength
Abrasions Resistance (ASTM D-1175-64T)

*Triton X-100 is a trademark for an alkylarylpolyether alcohol surfactant sold by Rohm & Haas Co., Philadelphia, Pa.

**Lubritron KN is a trademark for a softener comprised of a nonionic emulsion of a high density olefin sold by Chas. S. Tanner Co., Warwick, R.I.

***Catalyst-KR is a magnesium chloride-based catalyst solution sold by Sun Chemical Co., Wood River Junction, R.I.

The following table shows the pick-up properties of each of the controls as compared with compound (C), the product of Example II, above;

TABLE II

	RESIN ADD-ON			
	% Wet Pickup	% Wet Pickup	% Dry Pickup (after 1 wash)	% Dry Pickup (after 21 washes)
Aerotex 82	78	7.0	5.4	3.1
Permafresh 113 B	78	8.7	6.5	3.5
Product of Ex. II	75.5	8.0	3.5	2.5

Wrinkle recovery characteristics of the fabric of Example III, above, treated with the compounds of Table II is determined by a well-known method: ASTM D-1295-67-Warp direction only. The results of these tests follow:

TABLE III

	WRINKLE RECOVERY		
	Recovery in Degrees After 20 Initial Wash Cycles		
Blank (Untreated 50/50 PE:Cotton)	107	120	
CONTROLS { Aerotex 82	142	142	
Product of Ex. II	145	144	
	143	145	

Cantilever stiffness of the fabric of Example III using the compounds of Table II is determined using the method: ASTM D-1388-64-Warp direction only. The results are as follows:

TABLE IV

10	CANTILEVER STIFFNESS		
	Initial	Flexural Rigidity (mg-cm.) After 20 Wash Cycles	
Blank (Untreated 50/50 PE:Cotton)	79.95	67.58	
CONTROLS { Aerotex 82	88.68	62.17	
Product of Ex. II	89.43	55.94	
	96.65	67.07	

Tearing strength of the fabric of Example II, above, using the compounds of Table II is determined by the method: ASTM D-2261-64T-Warp direction only. The results are as follows:

TABLE V

25	TEARING STRENGTH BY THE TONGUE (SINGLE RIP) METHOD		
	Initial	Break Load (in pounds) After 20 Wash Cycles	
Blank (Untreated 50/50 PE:Cotton)	3.14	4.08	
CONTROLS { Aerotex 82	3.62	3.69	
Product of Ex. II	3.75	4.13	
	3.65	4.07	

Conventional soil redeposition test showed the product of Example II, i.e., compound (B), to be superior to Permafresh 113B (DMDHEU). Using a small amount of anti-soil-redeposition agent for polyester/cotton blends when subjecting the fabric of Example II, treated as taught in the same Example, to an otherwise conventional soil redeposition test showed the fabric treated with 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine/formaldehyde compound (B) performed as well as the Aerotex 82 treated fabric and better than the Permafresh 113B-treated fabric under the same conditions. In other words, the Aerotex 82-treated fabric and the compound (B)-treated fabric both remained almost completely white. The anti-soil-redeposition agent used is a water-soluble methyl cellulose derivative (4000 cps) bearing the trademark METHOCEL-90-HG and sold by Dow Chemical Corp., Midland, Michigan. A concentration of 1%, by weight, of Methocel-HG is added to the conventional soil redeposition test soiling bath alluded to hereinabove; the concentration, viz., 1% by weight, is based on the total weight of the soil bath. The use of methyl cellulose derivatives (Methocel) is claimed in copending application Ser. No. 22,140, filed Apr. 2, 1970, by A. S. Forschirm et al and now Pat. No. 3,668,000.

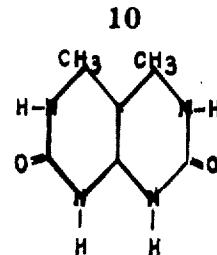
As is evident from Tables III and IV, above, the wrinkle recovery and stiffness performance of compound (B) compares very favorably with the performance of the commercial resins, even after multiple launderings. This indicates, in the case of wrinkle recovery, good bonding of the compound (B) resin to the fabric. Tear strength data reported in Table V, above, showed compound (B), like the commercial controls, produced no

significant deterioration in fiber strength. Abrasion resistance tests (ASTM D-1175-64T) not tabulated hereinabove, likewise indicated no significant deterioration in fiber strength.

Pursuant to statutory requirements, there are described above the invention and what are now considered its best embodiments. It should be understood, however, that the invention can be practiced otherwise than as specifically described, within the scope of the appended claims.

We claim:

1. A process for imparting a permanent press finish to a cellulosic fabric wherein the fabric is contacted with an aqueous solution of an aminoplast material, said aminoplast material being a product of the reaction of



with between about 2.8 and about 5.0 molar proportions of formaldehyde;

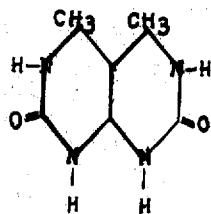
2. conducting step (1) in an aqueous medium;
3. conducting step (1) in the presence of a catalytic amount of an acid or base; and
4. conducting step (1) at a temperature between ambient temperature and the reflux temperature of the aqueous medium to effect reaction between said depicted compound and formaldehyde.

20 4. A process for imparting a permanent press finish to a cellulosic fabric comprising:

- a. contacting the fabric with an aqueous solution of an aminoplast material;
- b. conducting step (a) in the presence of an acid curing catalyst; and
- 25 c. subsequently exposing the fabric treated in step (b) to a temperature of at least about 275°F. to cure the aminoplast material, said aminoplast material being formed by
 1. contacting a molar proportion of initially crystalline 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine with between about 2.80 and about 5.0 molar proportions of formaldehyde;
 2. conducting step (1) in an aqueous medium;
 3. conducting step (1) in the presence of a catalytic amount of an acid or base; and
 4. conducting step (1) at a temperature between ambient temperature and the reflux temperature of the aqueous medium to effect reaction between the 2,7-dioxo-4,5-dimethyl-decahydropyrimido-[4,5-d]-pyrimidine and formaldehyde.

30 5. The process according to claim 4 wherein: the concentration of the aminoplast material in the aqueous solution used in step (a) is between about 2 percent and about 65 percent by weight; the acid curing catalyst used in step (b) is selected from the group consisting of inorganic salts which behave as latent acid catalysts; and the temperature employed in step (c) is between about 300°F. and about 350°F.

* * * * *



and a molar excess of formaldehyde, and reaction with the fabric is effected at an elevated temperature in the presence of an acidic catalyst

35 2. The process of claim 1 wherein the aminoplast is tetramethylolated.

3. A process for imparting a permanent press finish to a cellulosic fabric comprising:

- a. contacting the fabric with an aqueous solution of an aminoplast material;
- b. conducting step (a) in the presence of an acid curing catalyst; and
- 40 c. subsequently exposing the fabric treated in step (b) to an elevated temperature to cure the aminoplast material, said aminoplast material being formed by
 1. contacting a molar proportion of a compound having the formula

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