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(54) Durable press resin compositions.

(57) A durable press resin composition comprising (1) an aminoplast resin, (2) an aldehyde, and (3) a diluent. The durable press resin composition is combined with an acid catalyst and applied to textile materials to impart softness, improved wetting properties and durable press properties.

The durable press resin composition is applied at lower than normal dry add-on levels to textile materials to provide textile materials having lower levels of formaldehyde.

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DURABLE PRESS RESIN COMPOSITIONS

The present invention relates to compositions for treating textile materials and more particularly to durable press resin compositions for treating textile materials and to a process for treating textile materials to impart durable press finishes thereto.

Background of the Invention

Thermosetting durable press resins, also known as aminoplast resins have been used to treat textile materials to impart durable press properties and dimensional stability characteristics to the treated textile materials. These durable press resins, such as methylolated ureas or methylolated urea based derivatives which are obtained from the reaction of formaldehyde and urea or urea based derivatives generally contain from 95.8 to 97.8 weight percent of aminoplast resin and from 4.17 to 2.2 weight percent of formaldehyde.

Textile materials finished with durable press resins generally release formaldehyde due to the hydrolysis of unreacted methylol groups. The release of formaldehyde causes unpleasant odors and is a suspected health hazard. Therefore, it is preferred that textile materials finished with durable press resins be free of formaldehyde or at least have very low levels of formaldehyde.

Aminoplast resins have been developed which are free of formaldehyde and methylol groups; however, they do not impart satisfactory durable press properties to textile materials treated therewith. Surprisingly, it has been found that textile materials treated with an aminoplast resin and an aldehyde provides a durable press resin finish at substantially lower add-on levels with desirable properties. The resultant

textile material releases low levels of formaldehyde, has satisfactory durable press properties and exhibits dimensional stability.

Therefore, it is an object of this invention to provide durable press resin compositions for treating textile materials. Another object of this invention is to provide durable press finishes which impart softening and durable press properties to textile materials. A further object of this invention is to provide a textile material having low formaldehyde levels, a soft hand, durable press properties and dimensional stability characteristics.

Summary of the Invention

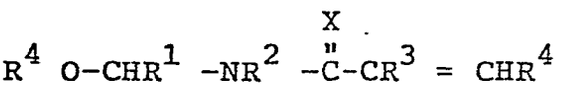
The foregoing objects and others which will become apparent from the following description are accomplished in accordance with this invention, generally speaking, by providing a durable press resin composition containing (1) from 0.25 to 45 percent by weight of an aminoplast resin, (2) from 0.03 to 37 percent by weight of an aldehyde, and (3) from 18 to 99.72 percent by weight of a diluent based on the weight of the durable press resin composition. The durable press resin composition is combined with an acid catalyst and applied to textile materials to impart durable press and dimensional stability properties thereto. In addition to providing improved durable press properties and dimensional stability characteristics, the treated textile materials have a low level of formaldehyde and a soft hand.

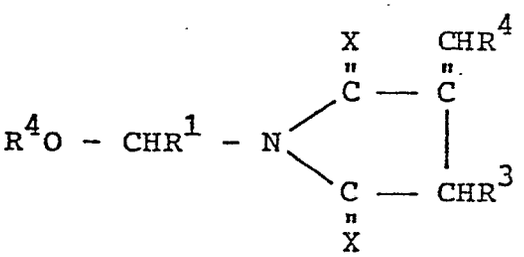
Detailed Description of the Invention

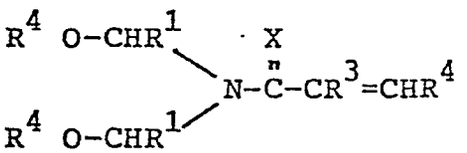
The aminoplast resins which are used in the compositions of this invention are well known in the art. Suitable examples of aminoplast resins are the urea formaldehydes, e.g., propylene urea formaldehyde, and dimethylol urea formaldehyde; melamine formaldehyde, e.g., tetramethylol melamines, and pentamethylol melamines; ethylene ureas, e.g., dimethylol ethylene urea, dihydroxy dimethylol ethylene urea, ethylene urea formaldehyde, hydroxy ethylene urea formaldehyde; carbamates, e.g., alkyl carbamate formaldehydes; formaldehyde-acrolein condensation products; formaldehyde-acetone condensation products; alkylol amides, e.g., methylol formamide, methylol

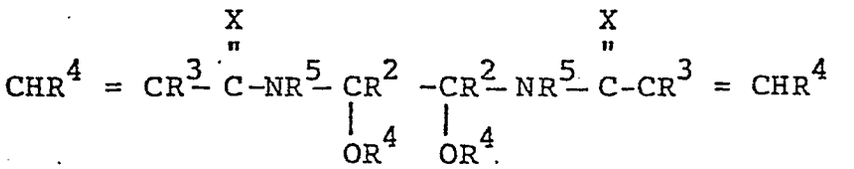
acetamide; acrylamides, e.g., N-methylol acrylamide, N-methylol methacrylamide, N-methylol-N-methacrylamide, N-methylmethylol-acrylamide, N-methylol methylene-bis(acrylamide), methylene-bis (N-methylol acrylamide); chloroethylene acrylamides; diureas, e.g., trimethylol acetylene diurea, tetramethylol-acetylene diurea; triazones, e.g., dimethylol-N-ethyl triazone, N,N'-ethylene-bis dimethylol triazone, halotriazones; haloacetamides, e.g., N-methylol-N-methylchloroacetamide; urons, e.g., dimethylol uron, dihydroxy dimethylol uron; and the like. Mixtures of two or more aminoplast resins may be used in the compositions of this invention.

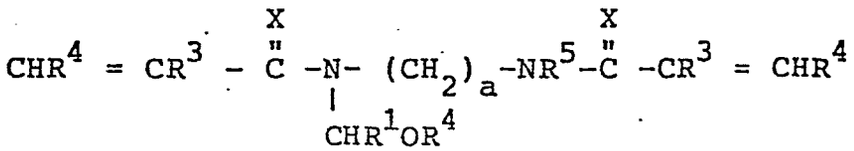
Other aminoplast resins which may be used in the compositions of this invention may be represented by the formulas:

(1) 

(2) 

(3) 

(4) 

(5) 

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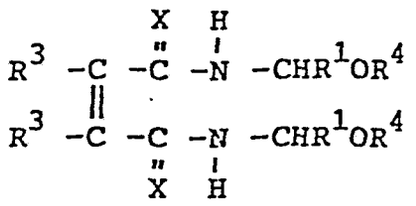

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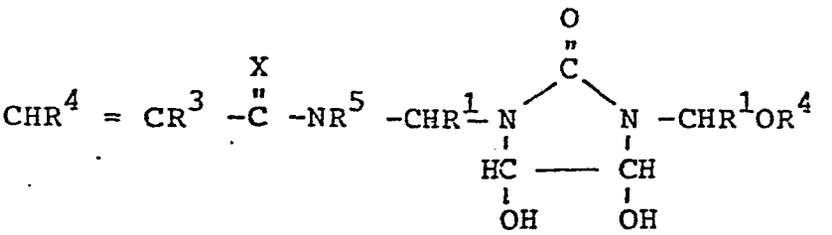

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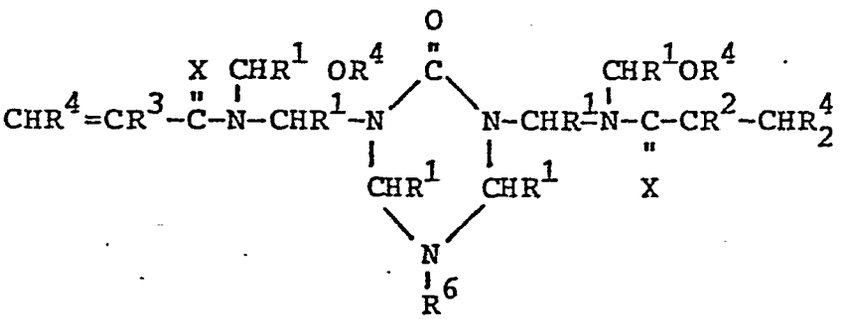

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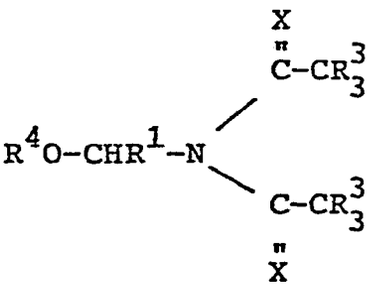

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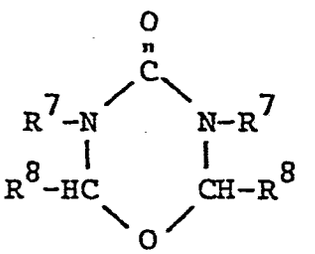
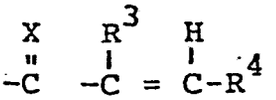


wherein R^1 is hydrogen, a lower alkyl radical or a radical from a saturated or unsaturated aldehyde, R^2 is hydrogen, a lower alkyl radical or a radical represented by the formula



R^3 is hydrogen or a methyl radical, R^4 is hydrogen or a lower alkyl radical, R^5 is hydrogen, a lower alkyl radical or CHR^1OR^4,

with at least one R^5 being CHR^1OR^4, R^6 is a lower alkyl radical or hydroxy alkyl radical, R^7 is hydrogen, hydroxy radical, or lower alkyl radical, R^8 is hydrogen, a lower alkyl radical, an alkylol radical, or an alkenol radical, X is oxygen or sulfur, and a is a number of from 1 to 6. Sulfur containing groups such as

$$-S-,\ -\overset{\overset{O}{\|}}{S}-,$$ or sulfonium may be substituted for the $-\overset{\overset{X}{\|}}{C}-$ group.

Suitable examples of aldehydes which may be used in the compositions of this invention are saturated and unsaturated aliphatic aldehydes having from 1 to 20 carbon atoms, such as formaldehyde, ethanal, propanal, propenal, propynal; isomers of butanal, pentanal, hexanal, heptanal, octanal, nonanal, decanal, undecanal, dodecanal, tridecanal, tetradecanal, pentadecanal, hexadecanal, heptadecanal, octadecanal, nonadecanal, ecosanal, butenal, hexenal, undecenal, furfural and the like. Other examples of substituted saturated and unsaturated aldehydes having from 1 to 20 carbon atoms are haloalkanals, such as chloroethanal, dichloroethanal, bromal, chloral, 2-bromopropanal, 2-chloropropanal, 3-chloropropanal, 2-chloro-2-methylpropanal, 2,3-dibromopropanal, 2,3-dichloropropanal, 2,2,3-trichloropropanal, 4-chlorobutanal, 2,3-dichlorobutanal, 2,2,3-trichlorobutanal and the like; hydroxyalkanals such as glycolaldehyde, 2,3-dihydroxypropanal, 3-hydroxybutanal, 4-hydroxypentanal, 3-hydroxy-2-methylpentanal and the like; alkylalkanals such as 2,2-dimethylpropanal, 2-ethylbutanal, 2-methylbutanal, 3-methylbutanal, 2-ethylhexanal, and the like; alkoxyalkanals such as ethoxyethanal, methoxyethanal and the like; oxoalkanals such as glyoxal, methylglyoxal, 2-phenoxypropanal, 4-methyl-2-oxopentanal, 2-oxopentanal, 4-oxopentanal and the like; haloalkenals such as 2-chloropropenal, 2-chlorobutenal and the like; and alkoxyalkenals such as 3-ethoxybutenal. Examples of aromatic substituted or unsubstituted aldehydes are benzaldehyde, tolualdehydes, salicylaldehyde, 1-phenylpropynal, 2-benzylidenebutanal, 2-benzylideneheptanal, hydroxybenzaldehydes, anisaldehyde, vanillin, piperanal, cinnamaldehyde, carboxybenzaldehydes and the like.

Diluents which are employed in the compositions of this invention are water and aliphatic alcohols having up to 8 carbon atoms.

A softening agent may be incorporated in the compositions of this invention to impart a soft hand to textile materials treated therewith. The term softening agent includes any material

which may be combined with the composition of this invention to impart a soft hand to treated textile materials.

Softening agents which may be added to the compositions of this invention are well known in the art. Examples of suitable softeners are organopolysiloxanes which are capable of being crosslinked. The crosslinkable organopolysiloxane compositions contain organopolysiloxanes having the general formula

$$HO\left(\begin{array}{c}R^{10}\\ |\\ SiO\\ |\\ R^{10}\end{array}\right)_dH$$

and a crosslinking agent such as a silane having the general formula

$$R^{10}_mSi(Y)_{4-m}$$

or siloxanes having -Si-O-Si- linkages and the remaining valences of the silicon atoms are satisfied by R^{10} and Y, in which R^{10}, which may be the same or different, is as defined above, Y is an acyloxy, oximo, alkoxy, aryloxy, halogen, aminoxy, amido or phosphato group, d is a number of from 1 to 1000, and m is 0 or 1. Catalysts such as those containing carboxylic acid salts of tin, zirconium, or titanium may be used with the organopolysiloxanes to promote crosslinking.

Further examples of softening agents which may be added to the compositions of this invention are non-crosslinkable polydiorganosiloxanes having the general formula

$$R^{10}_3SiO\left(\begin{array}{c}R^{10}\\ |\\ SiO\\ |\\ R^{10}\end{array}\right)_dSiR^{10}_3$$

in which R^{10} and d are the same as above.

Other softening agents which may be used in the composition of this invention are those obtained from the addition of silicon-bonded hydrogen atoms present in organopolysiloxanes to silicon-bonded aliphatically unsaturated groups present in other organopolysiloxanes. Organopolysiloxanes containing silicon-bonded hydrogen may be represented by the general formula

$$R^{10}_eH_fSiO_{\frac{4-e-f}{2}}$$

in which R^{10} is as defined above, e has a value of from 1.0 to 2.5, f has a value of from 0.005 to 2.0 and the sum of e + f is equal to from 1.005 to 3.0.

The organopolysiloxane containing silicon-bonded hydrogen may also be copolymers containing at least one unit per molecule of the formula

$$R^{10}_{e'}H_{f'}SiO_{\frac{4-e'-f'}{2}}$$

with the remaining siloxane units of the organopolysiloxane having the average formula

$$R^{10}_{e}SiO_{\frac{4-e}{2}}$$

where R^{10} and e are the same as above, e' has a value of from 0 to 2, f' has a value of from 1 to 2, and the sum of e' + f' is equal to from 1.0 to 3.0.

Generally, the copolymers contain from 0.5 to 99.5 mole percent of the siloxane units of the formula

$$R^{10}_{e'}H_{f'}SiO_{\frac{4-e'-f'}{2}}$$

and from 0.5 to 99.5 mole percent of the siloxane units of the formula

$$R^{10}_{e}SiO_{\frac{4-e}{2}}$$

where R^{10}, e, e' and f' are the same as above.

The organopolysiloxanes containing silicon-bonded aliphatically unsaturated groups may be represented by the formula

$$R^{10}_{e}R^{12}_{f}SiO_{\frac{4-e-f}{2}}$$

where R^{10}, e and f are the same as above and R^{12} represents a silicon-bonded aliphatically unsaturated group such as a vinyl or allyl radical. These organopolysiloxanes containing aliphatically unsaturated groups may also be copolymers having siloxane units of the formula

$$R^{10}_{e'}R^{12}_{f'}SiO_{\frac{4-e'-f'}{2}}$$

where R^{10}, R^{12}, e', f' and the sum of e' + f' are the same as above.

Generally, the copolymers contain from 0.5 to 99.5 mole percent of units having the formula

$$R^{10}_{e'}R^{12}_{f'}SiO_{\frac{4-e'-f'}{2}}$$

and from 0.5 to 99.5 mole percent of units having the formula

$$R^{10}_{e}SiO_{\frac{4-e}{2}}$$

where R^{10}, R^{12}, e, e' and f' are the same as above.

Any catalyst capable of promoting the addition of silicon-bonded hydrogen to silicon-bonded aliphatically unsaturated groups may be used in preparing these softeners. Preferably, the catalyst is platinum or a platinum compound or complex.

The silicone softeners may also contain organofunctional groups. Examples of such softeners are copolymers of aminofunctional polysiloxanes containing units of the formula

$$NH_2-R^{13}-(NHR^{13})_g\ SiO_{1.5}$$

and units of the formula

$$R^{10}_{e}SiO_{\frac{4-e}{2}}$$

wherein R^{10} and e are the same as above, R^{13} which may be the same or different is a divalent hydrocarbon radical having from 1 to 10 carbon atoms and g = 0, 1, or 2.

Other softeners which may be used are copolymers of ureidofunctional polydimethylsiloxanes having units of the formula

$$H_2N-\overset{O}{\overset{\|}{C}}-NH-R^{13}-SiO_{1.5}$$

and units of the formula

$$R^{10}_{e}SiO_{\frac{4-e}{2}}$$

where R^{10} and R^{13} are the same as above.

Softeners containing mercaptofunctional groups are polysiloxanes having units of the formula

$$(H-S-R^{13})_h\ SiO_{1.5}$$

and units of the formula

$$R^{10}_{e}SiO_{\frac{4-e}{2}}$$

wherein R^{10}, R^{13} and e are the same as above and h is a number of from 1 to 3.

Other organofunctional silicone softeners which may be included in the compositions of this invention are the silylated polyethers described in U. S. Patents Nos. 4,312,993 and 4,331,797 to Martin which are incorporated herein by reference.

Also, compositions containing silanol terminated polyorganosiloxanes and the silylated polyethers described in U. S. Patents Nos. 4,312,993 and 4,331,797 to Martin can be used as softeners in the compositions of this invention.

Another class of silicone softeners which may be included in the durable press compositions of this invention are those described in U. S. Patent No. 4,184,004 to Pines, which is incorporated herein by reference. These softeners consist of organosilicone terpolymers containing a plurality of reactive epoxy groups and a plurality of polyoxyalkylene groups. These organosilicone terpolymers may be prepared by the platinum catalyzed addition of an ethylenically unsaturated epoxy compound and an ethylenically unsaturated polyoxyalkylene organic radical free of olefinic unsaturation, preferably an alkyl compound, to hydrosiloxanes.

Organic softening agents may also be used in the durable press resin compositions of this invention in the presence or absence of the above silicone softeners. Suitable examples of organic softeners are fatty amides, fatty acid amines, and fatty acid amido amines; amido amines with mono- and diglycerides, quaternized ethoxylated fatty acid amines, hydroxyethyldiethylammonium sulfate and stearic quaternary ammonium compounds; fatty acid esters such as stearates, glycerol stearates, diethylene glycol stearates, and sulfonated fatty acid esters of polyethylene glycols and diethylene glycols; oxyalkylene polymers such as oxyethylene polymers, oxypropylene polymers, and copolymers thereof, salts of long-chain alcohols and fatty alcohol/fatty acid amide blends; fatty acids such as lauric, myristic, palmitic, oleic, and stearic acids; diethylene dipropyl benzoates; polyethylene polymers and sodium hydrocarbon sulfates. The softening

agent may be added directly to the durable press resin composition, or they may be emulsified or dissolved in water or organic solvents and then added to the resin composition.

When the softening agent is added to the durable press resin composition, i.e., an aminoplast resin, aldehyde and diluent, it is preferred that it be present in an amount such that the resultant finishing bath used for treating textile materials will contain up to about 8 parts by weight of softening agent. The softening agent may be dissolved in aliphatic alcohols such as methanol, ethanol, butanol, hexanol and octanol.

The durable press resin compositions of this invention, may be prepared by mixing the aminoplast resin, aldehyde and diluent in any order and at temperatures ranging from about 10°C to 90°C.

The durable press resin composition may be applied to any textile materials. Examples of suitable textile materials are cotton, rayon, polyester, polypropylene, polyethylene, polyurethane, polyamide, wool, hemp, natural silk, cellulose acetate and polyacrylonitrile fibers as well as mixtures of these fibers. The textile materials may consist of staple or monofilament fibers and fabrics made thereof.

The durable press resin compositions of this invention may be applied to the textile materials by any means known in the art, such as by spraying, immersion, foaming, padding, calendering or by gliding the fibers across a base which has been saturated with the compositions of this invention.

A preferred method for treating textile materials is to use a finishing bath containing a solution, dispersion or emulsion of the durable press resin compositions of this invention. The finishing bath composition contains the durable press resin composition, acid catalyst and softener, if desired. Also, the finishing bath composition may be further diluted with a diluent. It is preferred that the diluent be the same diluent or at least a diluent which is compatible with the diluent used in the durable press resin composition. Preferably the diluent is water or an aliphatic alcohol having from 1 to 8 carbon atoms.

The finishing bath preferably contains from 0.1 to about 99 parts by weight, and more preferably from about 5 to 50 parts by weight of durable press resin composition and from about 0.5 to 20 parts by weight, and more preferably from about 1 to 10 parts by weight of acid catalyst. The amount of diluent added to the finishing bath may range from 0 to 99.4 parts by weight and more preferably from about 10 to 75 parts by weight and the amount of softening agent, when present, may range from about 0 to about 8 parts by weight and more preferably from about 1 to 5 parts by weight.

When the durable press resin compositions are used in the form of an emulsion, any of the known surfactants can be used as emulsifying agents, including the anionic, cationic, nonionic and amphoteric surfactants.

Suitable examples of acid catalysts which may be used in the compositions of this invention are water soluble metal salts such as magnesium chloride, magnesium nitrate, magnesium sulfate, magnesium dihydrogenphosphate, zinc nitrate, zinc chloride, zinc tetrafluoroborate, aluminum chlorohydrate, aluminum chloride and mixtures of two of the above salts; water soluble ammonium and amine salts such as ammonium chloride, ammonium sulfate, aminomethylpropanol hydrochloride and aminomethylpropanol nitrate; ammonium and amine salts in combination with the metal salts described above; acids such as oxalic acid, gluconic acid, phosphoric acid, tartaric acid, maleic acid, p-toluenesulfonic acid and acetic acid; and combinations of the above acids with the above described metal salts.

The aminoplast resin component and the water soluble acid catalyst component should be kept separate until ready for use due to the instability of the mixture. The other components of this invention may be combined together in any order. It is, however, preferred that the other components be added to the aminoplast resin component.

The amount of the durable press resin composition of this invention which is applied to the textile material depends on the desired properties of the treated material. Generally, it is preferred that the textile material be treated with from 0.1 to 25 percent by weight of durable press resin composition, and more preferably from 0.2 to 20 percent by weight of the durable press resin composition, based on the weight of the

textile material.

The textile material finished with the composition of this invention is heated at an elevated temperature, e.g., from 80° to 200°C for a brief period of time; e.g., from 20 seconds to 15 minutes. Alternatively, the treated textile material can be dried below the above temperature range, e.g., from 50 to 95°C for a brief period of time, e.g., from 1 to 10 minutes, and then cured at an elevated temperature, e.g., from 125 to 200°C for an even briefer period of time, e.g., 15 to 60 seconds.

Textile materials treated with the durable press resin compositions of this invention exhibit all properties common to textile materials treated heretofore with aminoplast resins. In addition, when the levels of aminoplast resin are reduced in conventional systems, in order to lower the formaldehyde levels on the fabric, poor durable press properties and dimensional stability characteristics are observed. However, in the present invention, the addition of aldehydes permit a reduction of the aminoplast resin component of from 57 to 95 percent without adversely increasing the amount of formaldehyde present on the textile material after treatment, and without affecting the durable press properties and dimensional stability characteristics.

The addition of softener to the durable press resin compositions of this invention does not alter the durable press properties and dimensional stability characteristics of the fabric nor formaldehyde levels on the textile material. Textile materials treated with the durable press resin/softener compositions of this invention have a softer hand than those treated with the durable press resin compositions alone or with other conventional aminoplast resins. Furthermore, because the amount of aminoplast resin component required in the present invention to obtain durable press properties is significantly less than that required heretofore, the durable press finish is significantly more economical.

Other substances which may be incorporated in the composition of this invention are agents which improve abrasion resistance of the treated fibers, materials which improve the fragrance of the treated textile materials, antistatic agents, lubricants, fire retardant agents, soil resistant materials,

other hydrophilic, oleophilic, or hydrophobic agents and soil release materials such as those described in U. S. Patents Nos. 3,595,141 and 3,377,249 to Marco.

Specific embodiments of this invention are further illustrated in the following examples in which all parts and percentages are by weight unless otherwise specified. The amount of formaldehyde present on the treated textile materials is determined in accordance with the procedure described in the Technical Manual of the American Association Of Textile Chemists And Colorists (AATCC - Test No. 112-1978). The dimensional stability and durable press ratings are determined in accordance with AATCC test method number 135-1978 and 124-1978, respectively.

Example 1

Several compositions are prepared by dispersing the ingredients shown in Table I in water. The compositions are padded onto samples of polyester/cotton (65/35) fabric at 50 percent wet pick-up. The fabric is dried for 60 seconds at 120°C and cured for 20 seconds at 204°C. The treated fabric is then evaluated for: (a) parts per million formaldehyde; (b) dimensional stability through five home launderings; (c) durable press properties through five home launderings.

The results show that formaldehyde enhances the durable press ratings and dimensional stability of the fabric through multiple home launderings. Furthermore, the treated fabric contained less than 200 ppm (parts per million) formaldehyde. Generally, the textile industry requires 500 ppm or less.

TABLE I

Effect On Durable Press And Dimensional Stability With The Addition Of Formaldehyde To the Pad Bath

Dimethylol-dihydroxy ethylene-urea (45% aqueous solution) (Percent)	Formaldehyde (37% aqueous solution) (Percent)	Magnesium Chloride-Aluminum Chloride (30% aqueous solution of $MgCl_2$-$AlCl_3$ 9:1) (Percent)	Zinc Nitrate (25% aqueous solution) (Percent)	H_2O (Percent)	Fifth Wash Durable Press Rating	Fifth Wash Dimensional Stability (Percent)		Parts Per Million Formaldehyde
						Warp	Fill	
---	---	---	---	100	3	-2.8	-0.5	12
2.0	---	3.0	---	95.0	3	-1.8	-0.6	76
2.0	0.5	3.0	---	94.5	3.5	-1.3	-0.6	168
2.0	0.5	---	6.0	91.5	3.5	-1.6	-0.5	48

Example 2

Several compositions are prepared by dispersing the ingredients listed in Table II in water. These compositions are padded onto polyester/cotton (65/35) fabric at 50 percent wet pick-up. The fabric is dried and cured in accordance with the procedure described in Example I. The treated fabric is then evaluated for (a) parts per million formaldehyde; (b) dimensional stability through five home launderings; and (c) durable press rating through five home launderings.

The results show that the presence of formaldehyde in a durable press finish composition enhances the durable press ratings and dimensional stability of the fabric through multiple home launderings while remaining below 500 ppm of formaldehyde.

TABLE II

Effect On Durable Press And Dimensional Stability Using Various Resins With Formaldehyde In The Pad Bath

2-methoxyethyl carbamate (46% aqueous solution) (Percent)	alkylated urea-formaldehyde (48% aqueous solution) (Percent	Formaldehyde (37% aqueous solution) (Percent)	Magnesium chloride-Aluminum chloride (30% aqueous solution $MgCl_2$-$AlCl_3$ 9:1) (Percent)	H_2O (Percent)	Fifth Wash Durable Press Rating	Fifth Wash Dimensional Stability Warp (Percent)	Fifth Wash Dimensional Stability Fill (Percent)	Parts Per Million Formaldehyde
---	---	---	---	100.0	3	-2.8	-0.5	12
---	2.0	---	3.0	95.0	3.5	-1.8	-0.6	105
2.0	---	---	3.0	95.0	3	-1.8	-0.7	44
---	2.0	0.5	3.0	94.5	3.5	-1.4	-0.3	336
2.0	---	0.5	3.0	94.5	3.5	-1.6	-0.5	66

Example 3

Several compositions are prepared by dispersing the ingredients listed in Table III in water. These compositions are padded onto polyester/cotton (65/35) fabric at 50 percent wet pick-up. The fabric is dried and cured in accordance with the procedure described in Example 1. The treated fabric is then evaluated for (a) parts per million residual formaldehyde; (b) dimensional stability through five home launderings; and (c) durable press through five home launderings.

The results show that aldehydes enhance the durable press ratings and dimensional stability of the fabric through multiple home launderings while the formaldehyde levels are acceptable to the textile industry. Also, the data shows that formaldehyde is the most effective aldehyde.

TABLE III

Effect On Durable Press And Dimensional Stability
With The Addition Of Various
Aldehydes To The Pad Bath

Dimethylol-dihydroxy-ethylene-urea (45% aqueous solution) (Percent)	Formalde-hyde (37% aqueous solution) (Percent)	Acetal-dehyde (37% aqueous solu-tion) (Percent)	Propional-dehyde (97% aqueous solution) (Percent)	Magnesium chloride-Aluminum chloride (30% aqueous solution $MgCl_2$-$AlCl_3$ 9:1) (Percent)	H_2O (Percent)	Fifth Wash Durable Press Rating	Fifth Wash Dimensional Stability Warp (Percent)	Fill (Percent)	Parts Per Million Formal-dehyde
---	---	---	---	---	100.0	3	-2.8	-0.5	12
2.0	---	---	---	3.0	95.0	3	-1.8	-0.6	76
2.0	0.5	---	---	3.0	94.5	3.5	-1.4	-0.2	101
2.0	0.66	---	---	3.0	94.34	3.5	-1.4	-0.5	106
2.0	---	0.5	---	3.0	94.5	3.5	-1.5	-0.5	59
2.0	---	---	0.2	3.0	94.8	3.5	-1.8	-0.5	215

Example 4

Several bath compositions are prepared by dispersing the ingredients listed in Table IV in a water-ethanol solvent. These formulations are padded onto polyester/cotton (65/35) fabric at a 30 percent wet pick-up. The fabric is dried and cured in accordance with the procedure described in Example 1. The treated fabric is then evaluated for (a) parts per million formaldehyde; (b) dimensional stability through five home launderings; and (c) durable press through five home launderings

The results show that the presence of an aromatic aldehyde in a water-ethanol solvent system will improve the durable press ratings and dimensional stability of the fabric through multiple home launderings. Also, the data indicates that the dimethyoldihydroxyethyleneurea contributes to the increased formaldehyde level.

TABLE IV

Effect On The Durable Press And Dimensional Stability
Using Salicylaldehyde In An Ethanol-Water Pad Bath

Dimethyloldihydroxyethyleneurea (45% aqueous solution) (Percent)	Salicylaldehyde (37% ethanolic solution) (Percent)	Magnesium Chloride-Aluminum Chloride (30% aqueous solution $MgCl_2$-$AlCl_3$ 9:1) (Percent)	H_2O (Percent)	Ethanol (Percent)	Fifth Wash Durable Press Rating	Fifth Wash Dimensional Stability Warp (Percent)	Fifth Wash Dimensional Stability Fill (Percent)	Parts Per Million Formaldehyde
---	---	---	100.0	---	3	-2.8	-0.5	12
---	---	---	5.5	94.5	3	-2.8	-0.5	5
2.0	---	3.0	0.5	94.5	3	-1.9	-0.1	129
2.0	0.5	3.0	0.3	94.2	3.5	-1.85	-0.3	132

Example 5

Several compositions are prepared by dispersing the ingredients of Table V in water. These compositions are padded onto 100 percent cotton at a 50 percent wet pick-up. The fabric is dried and cured in accordance with the procedure described in Example 1. The treated fabric is then evaluated for (a) parts per million formaldehyde; (b) dimensional stability through five home launderings; and (c) durable press through five home launderings.

The results show that a durable press composition containing formaldehyde and dimethyloldihydroxyethyleneurea substantially improves the dimensional stability and durable press properties of 100 percent cotton as compared with a durable press composition containing only dimethyloldihydroxyethyleneurea.

TABLE V

Effect On Durable Press And Dimensional Stability
On 100% Cotton Using A Solution Containing
Formaldehyde And A Dimethyloldihydroxyethyleneurea Resin

Dimethyloldihydroxy-ethyleneurea (45% aqueous solution) (Percent)	Formaldehyde (37% aqueous solution) (Percent)	Magnesium Chloride-Aluminum Chloride (30% aqueous solution ($MgCl_2$-$AlCl_3$ 9:1) (Percent)	H_2O (Percent)	Fifth Wash Durable Press Rating	Fifth Wash Dimensional Stability (Percent)		Parts Per Million Formaldehyde
					Warp	Fill	
---	---	---	100.0	1	-6.45	+0.35	4
4.0	---	4.0	92	2	-2.0	+0.25	103
4.0	0.50	4.0	91.5	2	-1.8	+0.4	247
5.0	---	4.0	91	2	-2.0	+0.2	357
5.0	0.25	4.0	90.75	2	-1.85	+0.4	192
5.0	0.50	4.0	90.5	2	-1.6	+0.1	374

Example 6

Several compositions are prepared by dispersing the ingredients listed in Table VI in water. These compositions are padded onto polyester/cotton (65/35) fabric at 50 percent wet pick-up. The fabric is dried and cured in accordance with the procedure described in Example 1. The treated fabric is then evaluated for (a) parts per million formaldehyde; (b) dimensiona stability through five home launderings; (c) durable press through five home launderings; and (d) fabric hand.

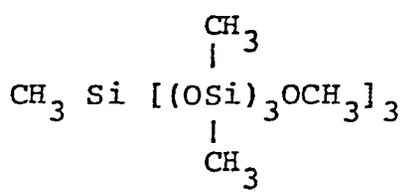
The results show that the presence of formaldehyde in a durable press finishing composition containing dimethyloldihydroxyethyleneurea improves the durable press ratings and dimensional stability of the fabric through multiple home launderings while the formaldehyde level is less than 300 ppm. In addition, the presence of the softener has no effect on the amount of formaldehyde present on the fabric, nor the durable press ratings and dimensional stability characteristics of the fabric through multiple launderings. Furthermore, all fabrics treated with softeners have a soft, silky hand. The results are shown in Table VI.

The softeners shown in Table VI are prepared in the following manner:

(a) A 33 percent aqueous emulsion of a softener is prepared by heating a mixture containing 124 parts of succinic anhydride and 2,278 parts of oxyethylene-oxypropylene triol copolymer, having a molecular weight of 6360 and a weight ratio of oxyethylene to oxypropylene of 7 to 3 at 120°C for eighteen hours in a reaction vessel. The resultant product is a yellow liquid having a viscosity of 4,168 cs. at 25°C and an acid content of 0.58 milliequivalents per gram (theoretical 0.5).

The resultant product is mixed with 238 parts by weight of aminopropyltriethoxysilane at 70°C for 3.0 hours. This reaction product is a yellow liquid having a viscosity of about 30,000 cs. at 25°C. The reaction product is mixed with 660 parts by weight of hydroxy terminated polydimethylsiloxane at 50°C for 6 hours. The resultant product is a white, opaque fluid having a viscosity of 1,500,000 cs. at 25°C. The product is then combined with 6,700 parts by weight of water. A white, opaque emulsion having a viscosity of 50 cs. at 25°C is obtaine

(b) A 40 percent aqueous emulsion consisting of 34 percent water, 16 percent of the polymer made in (a) and 50 percent of an emulsion containing 50 percent of a polysiloxane represented by the formula



(c) A 33 percent aqueous solution of a polymer is prepared by heating a mixture containing 150 parts of succinic anhydride and 2880 parts of oxyethylene-oxypropylene triol copolymer, having a molecular weight of 6360 and a weight ratio of oxyethylene to oxypropylene of 7 to 3, for eighteen hours at 120°C. The product is a yellow liquid having a viscosity of 4,168 mPa.s at 25°C, and an acid content of 0.58 milliequivalents per gram (theoretical 0.5 meq/g).

The resultant product is then mixed with 300 parts of aminopropyltriethoxysilane and heated at 70°C for 2 hours. The product is a yellow liquid having a viscosity of about from 30,000 mPa.s at 25°C. The resultant product is then mixed with 6670 parts of water to form a clear, straw-colored solution having a viscosity of 50 mPa.s at 25°C.

(d) A 25 percent active aqueous emulsion of a fatty acid condensation product.

(e) A 33 percent aqueous solution of a polymer is prepared by heating a mixture containing 124 parts of succinic anhydride, 930 parts of oxyethylene diol having a molecular weight of 1500 at 120°C for eighteen hours in a reaction vessel. The resultant product is a yellow liquid having an acid content of 1.2 milliequivalents per gram.

The resultant product is mixed with 374 parts of aminopropyltriethoxysilane at 70°C for 3.0 hours. The reaction product is mixed with 2702 parts of water. A clear straw-colored solution is obtained.

TABLE VI

Effect On Durable Press And Dimensional Stability
Using Formaldehyde In A Durable Press
Softener Treatment Bath

Examples	Softener (Percent)	Dimethylol-dihydroxy ethylene-urea (45% aqueous solution) (Percent)	Formaldehyde (37% aqueous solution) (Percent)	Magnesium Chloride-Aluminum Chloride (30% aqueous solution $MgCl_2$-$AlCl_3$ 9:1) (Percent)	H_2O (Percent)	Fifth Wash Durable Press Rating	Fifth Wash Dimensional Stability Warp (Percent)	Fifth Wash Dimensional Stability Fill (Percent)	Parts Per Million Formaldehyde
---	---	---	---	---	100.0	3	-2.8	-0.5	12
---	---	2.0	---	3.0	95	3	-1.8	-0.6	76
(a)	3.0	2.0	---	3.0	92	3	-1.9	-0.55	79
---	---	2.0	0.5	3.0	94.5	3.5	-1.3	-0.6	168
(a)	3.0	2.0	0.5	3.0	91.5	3.5	-1.5	-0.4	88
(b)	2.4	2.0	0.5	3.0	92.1	3.5	-1.4	-0.3	85
(c)	3.0	2.0	0.5	3.0	91.5	3.5	-1.8	-0.6	294
(d)	4.7	2.0	0.5	3.0	89.8	3.5	-1.4	-0.3	201
(a)	18.2	2.0	0.5	3.0	76.3	3.5	-1.6	-0.4	277
(e)	2.5	2.0	0.5	3.0	92.0	3.5	-1.5	-0.5	150

Example 7

Several compositions are prepared by dispersing the ingredients shown in Table VII in water. The softener is prepared in accordance with the procedure described in Example 6. These compositions are padded onto samples of polyester/cotton (65/35) fabric at a 50 percent wet pick-up. The fabric is dried and cured in accordance with the procedure described in Example 1. The treated fabric is then evaluated for (a) parts per million formaldehyde; (b) dimensional stability through five home launderings; (c) durable press through five home launderings; and (d) fabric hand. The results of these evaluations indicate that the presence of formaldehyde in a finishing bath containing varying levels of dimethyloldihydroxyethyleneurea enhance the durable press ratings and dimensional stability of the fabric through multiple home launderings while having less than 300 parts per million of formaldehyde. In addition, the presence of softener has no effect on the amount of formaldehyde present on the fabric nor the durable press ratings and dimensional stability characteristics of the fabric through multiple launderings. Furthermore, all fabrics treated with softeners have a soft, silky hand. The results are shown in Table VII.

TABLE VII

Effect On The Durable Press And Dimensional Stability
Using Formaldehyde In A Durable Press/Softener
Treatment Bath Containing
Dimethyloldihydroxyethyleneurea

Softener (a) (33% aqueous emulsion) (Percent)	Dimethyloldihydroxyethyleneurea (45% aqueous solution) (Percent)	Formaldehyde (37% aqueous solution) (Percent)	Magnesium Chloride-Aluminum Chloride (30% aqueous solution $MgCl_2$-$AlCl_3$ 9:1) (Percent)	H_2O (Percent)	Fifth Wash Durable Press Rating	Fifth Wash Dimensional Stability (Percent) Warp	Fifth Wash Dimensional Stability (Percent) Fill	Parts Per Million Formaldehyde
---	---	---	---	100.0	3	-2.8	-0.5	12
---	2.0	---	3.0	95.0	3	-1.8	-0.6	76
---	2.0	0.5	3.0	94.5	3.5	-1.3	-0.6	168
3.0	2.0	0.5	3.0	91.5	3.5	-1.5	-0.44	88
3.0	2.0	---	3.0	92.0	3	-1.9	-0.55	79
---	5.0	---	3.0	92.0	3.5	-1.55	-0.4	177
---	5.0	0.5	3.0	91.5	3.5	-1.05	-0.25	296
3.0	5.0	0.5	3.0	88.5	3.5	-1.2	-0.2	218
3.0	5.0	---	3.0	89.0	3.5	-1.45	-0.55	156

Example 8

Several compositions are prepared by dispersing the ingredients listed in Table VIII in water. The softener is prepared in accordance with the procedure described in Example 6. These formulations are padded onto samples of polyester/cotton (65/35) fabric at 50 percent wet pick-up. The fabric is dried and cured in accordance with the procedure described in Example 1. The treated fabric is then evaluated for (a) parts per million formaldehyde; (b) dimensional stability through five home launderings; (c) durable press through five home launderings; and (d) fabric hand. The results of these evaluations show that the presence of an aldehyde in the formulation can enhance the durable press ratings and dimensional stability of the fabric through multiple home launderings while having formaldehyde levels which are acceptable by the textile industry. Furthermore, all fabrics treated with softeners have a soft, silky hand. The results are shown in Table VIII.

TABLE VIII

Effect On The Durable Press And Dimensional Stability
Using An Aldehyde In A
Durable Press/Softener Treating Bath

Softener (a) (33% aqueous emulsion) (Percent)	Dimethyloldihydroxyethyleneurea (45% aqueous solution) (Percent)	Formaldehyde (37% aqueous solution) (Percent)	Acetaldehyde (37% aqueous solution) (Percent)	Magnesium Chloride-Aluminum Chloride (30% aqueous solution $MgCl_2$-$AlCl_3$ 9:1) (Percent)	H_2O (Percent)	Fifth Wash Durable Press Rating	Fifth Wash Dimensional Stability (Percent) Warp	Fill	Parts Per Million Formaldehyde
---	---	---	---	---	100.00	3	-2.8	-0.5	12
---	2.0	---	---	3.0	95.0	3	-1.8	-0.6	76
3.0	2.0	0.5	---	3.0	91.5	3.5	-1.5	-0.4	88
3.0	2.0	1.13	---	3.0	90.87	3.5	-1.25	-0.2	283
3.0	2.0	---	0.5	3.0	91.5	3.5	-1.5	-0.4	54
4.0	2.0	---	0.66	3.0	90.34	3.5	-1.7	-0.4	35

Example 9

Several compositions are prepared by dispersing the ingredients listed in Table IX in water. The softener is prepared in accordance with the procedure in Example 6. These formulations are padded onto polyester/cotton (65/35) fabric at 50 percent wet pick-up. The fabric is dried and cured according to the procedure in Example 1. The treated fabric is then evaluated for (a) parts per million formaldehyde; (b) dimensional stability through five home launderings; (c) durable press through five home launderings; and (d) fabric hand. The results of these evaluations show that the presence of formaldehyde in the formulation enhances the durable press ratings and dimensional stability of the fabric through multiple launderings while the fabric contains less than 250 parts per million of formaldehyde. In addition, all fabrics treated with softeners have a soft, silky hand. Furthermore, variation of the amount of acid catalyst present in the formulation has no effect on either of the above results. The results are shown in Table IX.

TABLE IX

Effect On The Durable Press And Dimensional Stability Using Formaldehyde In A Durable Press/Softener Treating Bath Containing Excess Acid Catalyst

Softener (a) (33% aqueous emulsion) (Percent)	Dimethyloldihydroxyethylene urea (45% aqueous solution) (Percent)	Formaldehyde (37% aqueous solution) (Percent)	Magnesium Chloride-Aluminum Chloride (30% aqueous solution $MgCl_2$-$AlCl_3$ 9:1) (Percent)	H_2O (Percent)	Fifth Wash Durable Press Rating	Fifth Wash Dimensional Stability (Percent) Warp	Fill	Parts Per Million Formaldehyde
---	---	---	---	100.0	3	-2.8	-0.5	12
---	2.0	---	3.0	95.0	3	-1.8	-0.6	76
3.0	2.0	0.5	3.0	91.5	3.5	-1.5	-0.4	88
3.0	2.0	0.5	5.0	89.5	3.5	-1.55	-0.35	233
3.0	2.0	0.5	10.0	84.5	3.5	-1.5	-0.20	143

Example 10

Several compositions are prepared by dispersing the ingredients shown in Table X in water. The softener is prepared in accordance with the procedure described in Example 6(a). These compositions are padded onto 100 percent cotton at 50 percent wet pick-up. The fabric is dried and cured in accordance with the procedure described in Example 1. The treated fabric is then evaluated for (a) parts per million formaldehyde; (b) dimensional stability through five home launderings; (c) durable press through five home launderings; and (d) fabric hand. The results show that the presence of formaldehyde in a durable press/softener finishing composition containing dimethyloldihydroxyethyleneurea improves dimensional stability and durable press properties of cotton in comparison to that observed utilizing durable press/softener compositions containing only dimethyldihydroxyethyleneurea. Furthermore, the fabric had a soft, silky hand. The results are shown in Table X.

TABLE X

Softener (a) (33% aqueous emulsion) (Percent)	Dimethyloldihydroxyethylene urea (45% aqueous solution) (Percent)	Formaldehyde (37% aqueous solution) (Percent)	Magnesium Chloride-Aluminum Chloride (30% aqueous solution $MgCl_2$-$AlCl_3$ 9:1 (Percent)	H_2O (Percent)	Fifth Wash Durable Press Rating	Fifth Wash Dimensional Stability Warp (Percent)	Fifth Wash Dimensional Stability Fill (Percent)	Parts Per Million Formaldehyde
---	---	---	---	100.0	1	-6.45	+0.35	4
---	4.0	---	4.0	92.0	2	-2.0	+0.25	103
---	4.0	0.5	4.0	91.5	2	-1.8	+0.4	247
3.0	4.0	0.5	4.0	88.5	2	-1.8	+0.6	202
---	5.0	---	4.0	91.0	2	-2.0	-0.2	357
---	5.0	0.5	4.0	90.5	2	-1.8	0	374
3.0	5.0	0.5	4.0	87.5	2	-1.6	+0.1	484
---	5.0	0.25	4.0	90.75	2	-1.85	+0.4	192
3.0	5.0	0.25	4.0	87.75	2	-1.75	+0.3	146

Claims:

1. A durable press resin composition containing (1) from 0.25 to 45 percent by weight of an aminoplast resin, (2) from 0.03 to 37 percent by weight of an aldehyde, and (3) from 18 to 99.72 percent by weight of a diluent based on the weight of the durable press resin composition.

2. The composition of claim 1, chracterized in that it contains addtionally a softening agent (4).

3. The composition of claim 1, characterized in that the aldehyde (2) is a saturated or unsaturated, unsubstituted or substituted aliphatic aldehyde having from 1 to 20 carbon atoms or an unsubstituted or substituted aromatic aldehyde.

4. The composition of claim 1 characterized in that the diluent (3) is water or an aliphatic alcohol having from 1 to 8 carbon atoms.

5. The composition of claim 2 characterized in that the softening agent (4) is a crosslinkable organopolysiloxane, containing an organopolysiloxane of the formula

$$HO\left[\begin{array}{c} R^{10} \\ | \\ SiO \\ | \\ R^{10} \end{array}\right]_d H$$

and a crosslinking agent selected from the group consisting of silanes of the formula

$$R^{10}_m\ Si(Y)_{4-m}$$

and siloxanes having Si-O-Si linkages wherein the unsatisfied valences of the silicon atoms are satisfied by R^{10} and Y, R^{10} is a monovalent hydrocarbon radical having from 1 to 20 carbon

atoms, Y is a hydrolizable group selected from acyloxy, oximo, alkoxy, aryloxy, halogen, aminoxy, amido and phosphate radicals, d is from 1 to 1000 and m is 0 or 1, or a non-crosslinkable organopolysiloxane of the formula

$$R_3^{10}-SiO\left[\begin{array}{c}R^{10}\\ |\\ SiO\\ |\\ R^{10}\end{array}\right]_d Si-R_3^{10}$$

where R^{10} and d are the same as above, or a silylated polyether, or a silylated polyether and a silanol containing organopolysiloxane or an organic softener.

6. A textile finishing bath containing from 0.1 to 99 parts by weight of the durable press resin composition of claim 1, from 0.5 to 20 parts by weight of an acid catalyst, from 0 to 99.4 parts by weight of diluent and from 0 to 8 parts by weight of softening agent.

7. The textile finishing bath of claim 6 wherein the diluent is water.

8. A process for coating textile materials with a durable press resin composition which comprises applying a composition containing (1) an aminoplast resin, (2) an aldehyde, (3) a diluent and an acid catalyst to a textile material, and thereafter drying the coated material at an elevated temperature.

9. A process for coating textile materials with a durable press resin composition which comprises applying a composition containing from 0.1 to 99 parts by weight of the durable press resin composition of claim 1, (2) from 0 to 20 parts by weight of acid catalyst, (3) from 0 to 99.4 parts by weight of diluent and (4) from 0 to 8 parts by weight of softening agent, to a textile material and thereafter drying

the coated material by heating to a temperature of at least 50°C and then increasing to a temperature of at least 125°C.

10. The coated textile material as prepared according to the process of claim 8 or 9.

0107799

European Patent Office

EUROPEAN SEARCH REPORT

Application number

EP 83 10 9683

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. ³)
X	CH-A- 351 938 (RADUNER) * Claims; page 1, lines 66-72; page 2, lines 1-3, 13-19; examples *	1-4,6-10	D 06 M 15/54 C 08 G 12/02 C 08 L 61/20
X,Y	FR-A-1 218 696 (CALICO) * Abstract; examples *	1-4,6-10	
X	EP-A-0 052 830 (BAYER AG) * Claims; page 4, lines 3-7 *	1-4,6-10	
X	CHEMICAL ABSTRACTS, vol. 85, no. 10, 6th September 1976, page 101, no. 64741e, Columbus, Ohio, US & JP - A - 76 40 498 (TOYOBO CO., LTD.) 05-04-1976 * Abstract *	1-4,6-10	TECHNICAL FIELDS SEARCHED (Int. Cl. ³)
Y	DE-A-2 543 978 (PFERSEE) * Claims *	1,5	D 06 M

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	12-01-1984	DERAEDT G.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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