United States Patent [19]

Reinhardt et al.

[11] **3,909,861**

[45] **Oct. 7, 1975**

[54] **ALUMINUM CHLORHYDROXIDE CATALYST SYSTEMS FOR TREATMENTS TO GIVE WRINKLE RESISTANT TEXTILES**

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[22] Filed: **Oct. 5, 1973**

[21] Appl. No.: **404,042**

[52] **U.S. Cl.** **8/184;** 8/185; 8/186; 8/187; 252/442; 252/463

[51] **Int. Cl.**[2] **D06M 13/34;** B01J 27/06; B01J 23/08

[58] **Field of Search** 8/184, 185, 186, 187; 252/442, 463

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[57] **ABSTRACT**

Aluminum chlorhydroxide catalyst systems are applicable in a broad variety of finishing techniques for producing wrinkle resistant textile materials. $Al_2(OH)_5Cl$ is unique in its versatile effectiveness in the catalysis of formaldehyde and formaldehyde-amide treatments of cellulose-containing textiles. It can function alone as catalyst, with strong acids to attenuate their catalytic activity, or with weak acids and salts to give synergistically activated catalysis.

14 Claims, No Drawings

ALUMINUM CHLORHYDROXIDE CATALYST SYSTEMS FOR TREATMENTS TO GIVE WRINKLE RESISTANT TEXTILES

FIELD TO WHICH INVENTION RELATES

This invention relates to wrinkle resistant fabrics. More specifically, it relates to catalyst systems that are useful in the treatment of cellulose-containing textile materials with formaldehyde and certain formaldehyde-amide adduct finishing agents to give wrinkle resistant textile products.

OBJECTS OF THE INVENTION

It is an object of the present invention to obtain fabrics with levels of wrinkle resistance that make them valuable for durable press clothing and household items.

It is a further object to provide catalyst systems that are efficient and practical for use in conventional as well as in specialized and unusual finishing treatments for cellulose-containing textiles.

A still further object is to provide a material that can be used with strong acid catalysts to dimish the undesirable effects of these acids without eliminating the excellent catalysis they produce.

A still further object is to provide a material that can be used with weak acids and salts to give catalytic effects greater than those of either component employed separately.

A still further object is also to provide catalyst systems which give durable press fabrics which though fully cured can be creased by simple processing methods so that the creases as well as the smoothness and wrinkle resistance are durable to repeated laundering.

Other objects will become apparent to those of ordinary skill in the art upon studying the specification and examples of the invention.

HOW THE OBJECTS OF THE INVENTION ARE ACHIEVED

The objects of the present invention are achieved by the use of catalyst systems based upon aluminum chlorhydroxide in treatments for cellulose-containing textiles with formaldehyde or foramldehyde-amide adduct finishing agents. These systems in which aluminum chlorhydroxide are used either alone or in combination with other materials provide efficient catalysis in conventional processing as well as in specialized and unusual treatments. Unexpectedly, aluminum chlorhydroxide functions in various roles depending upon the finishing treatment in which it is used. It acts alone as catalyst in many treatments, as a moderator with strong acids, and as an activator with weak acids and certain salts. Furthermore, fully cured, wrinkle resistant fabrics from treatments with aluminum chlorhydroxide catalysis can be durably creased by simple processing methods--methods so simple that they are applicable in the home.

ALUMINUM CHLORHYDROXIDE

Aluminum chlorhydroxide is widely used in cosmetics, deodorants, shaving lotions, and other personal products. Because of the large amounts consumed, the price is relatively low even in the purity required for these uses. Although various aluminum chlorhydroxide complexes are known, that commonly used and, thus readily available, has the empirical formula: $Al_2(OH)_5Cl$. It is sometimes called aluminum hydroxychloride or, the 5/6 basic aluminum chloride complex, as it may be considered to be aluminum chloride with 5/6 of the chlorine replaced with hydroxyl groups. Development of a grade suitable for textile processing, but not necessarily as pure as needed for cosmetics, could afford a further decrease in the cost of aluminum chlorhydroxide.

Specifications for the present commercial product indicate the presence of about two molecules of water of hydration per complex unit. Furthermore, there are indications that aluminum chlorhydroxide exists, particularly in aqueous solutions, as oligomers of the simple empirical formula which may be represented as: $[Al_2(OH)_5Cl.2H_2O]x$ wherein x may be 1 or an integer of low value.

Solutions of $Al_2(OH)_5Cl$ are mildly acidic and have excellent buffering capacity. Solutions ranging to 50 percent concentration exhibit a pH of about 4-4.5. By its usage for many years in cosmetic applications, aluminum chlorhydroxide has been demonstrated to be safe even for direct application to human skin.

Although $Al_2(OH)_5Cl$ is the most common of the aluminum chlorhydroxide complexes, other are known. They all may be represented by the simple formula: $Al_2(OH)_nCl_{6-n}$ wherein n is an integer below 6. Often the complexes exist in mixtures in which the overall composition is, for simplicity, expressed as the average formula rather than that indicative of the true structure. Thus, the formulae are sometimes expressed such that the value of n is not a whole number. For example, we have used a complex in which the empirical formula was $Al_2(OH)_{4.67}Cl_{1.33}$ although this probably was, in reality, a mixture with a ratio of two $Al_2(OH)_5Cl$ complexes to one $Al_2(OH)_4Cl_2$ complex.

The lower the value of n in the general formula for the aluminum chlorhydroxide complexes, the greater is the acidity of the complex and the greater is the catalytic power of the complex. However, as acidity and catalytic power become greater, the greater is the need for control of the finishing treatment to preclude degradation and other undesirable side effects to fabric.

SCOPE OF THE INVENTION

With suitable adjustment, all of the various aluminum chlorhydroxides can be used in the catalysis of wrinkle resistance finishing treatments. However, the most versatile is $Al_2(OH)_5Cl$. Those complexes in which the ratio of OH to Cl is lower are more acidic and thus less applicable in some embodiments of the invention. $Al_2(OH)_5Cl$ is the preferred complex for use in the catalyst systems of this invention.

Concentrations of $Al_2(OH)_5Cl$ that can be used are 0.1 to about 5 percent by weight of the treatment bath. Preferred concentration is from about 0.25 to 3 percent.

Aluminum chlorhydroxide can be employed alone as catalyst for wrinkle resistance treatments but in certain embodiments of the invention, $Al_2(OH)_5Cl$ is used in conjunction with other materials to produce specific catalytic effects. It has been found that when used in conjunction with strong acid catalysts, $Al_2(OH)_5Cl$ serves to buffer the treatment solution and attenuate the catalytic activity of the strong acid. On the other hand, when $Al_2(OH)_5Cl$ is used with weak acids and certain salts, a synergistic effect results to give catalyst systems with greater activity than that of either of the components individually. This ambivalent behavior in diverse catalyst systems is unexpected and surprising. It

makes aluminum chlorhydride a valuable chemical for broad application and utility in the catalysis of a wide range of wrinkle resistance treatments.

Among strong acids with which $Al_2(OH)_5Cl$ can be used to moderate their catalytic activity are the mineral acids, hydrochloric acid and nitric acid but not sulfuric acid (immediate precipitation occurs when $Al_2(OH)_5Cl$ is used with sulfuric acid) and sulfonic acids such as hydroxymethanesulfonic acid and the like. Concentrations operative in strong acid/$Al_2(OH)_5Cl$ catalyst systems include from about 0.1–1.2 percent acid and 1–3% $Al_2(OH)_5Cl$.

An entirely different catalytic effect is produced in treatments in which $Al_2(OH)_5Cl$ is used with weaker inorganic acids such as phosphoric acid and many organic acids. A synergistic interaction occurs so that greater catalysis results from the use of such acids and $Al_2(OH)_5Cl$ than from either alone. Among organic acids which have been found to give synergistically activated catalyst systems with $Al_2(OH)_5Cl$ are fatty acids such as formic acid, acetic acid, propionic acid, and the like, unsaturated acids such as acrylic acid and the like, dicarboxylic acids such as oxalic acid, succinic acid and the like, hydroxy-substituted acids such as glycolic acid, lactic acid, malic acid, citric acid and the like, halo-substituted acids such as chloroacetic acid, trichloroacetic acid and the like, and cyanosubstituted acids such as cyanoacetic acid and the like. For catalyst systems based on these acids, concentrations of $Al_2(OH)_5Cl$ of about 0.1-4% are used with about 0.1-5% of the acid.

Synergistic catalyst effects also are produced when $Al_2(OH)_5Cl$ is used in conjunction with certain salts such as magnesium chloride, zinc chloride, zinc nitrate, and the like. About 2% $Al_2(OH)_5Cl$ is used with about 0.2–3 percent of the salt although other concentrations of the aluminum complex probably could be employed. $Al_2(OH)_5Cl$ is not compatible with $Mg(H_2PO_4)_2$ nor with alkaline salts such as potassium carbonate, sodium bicarbonate, and the like.

The catalyst systems of this invention can be used in treatments of textiles with formaldehyde and formaldehyde-amide adduct finishing agents. There are advantages in the use of these catalyst systems with formaldehyde and with certain, but not all, formaldehyde-amide adduct agents. The catalyst systems of this invention operate well with urea, cyclic urea, uron, and carbamate type finishing agents. Typical of these agents are dimethylol urea, partially methylolated urea, methylated urea-formaldehyde, dimethylol ethyleneurea, dimethylol dihydroxyethyleneurea, dimethylol propyleneurea, dimethylol substituted propyleneurea, tetramethylol acetylenediurea, bis(methoxymethyl)uron, dimethylol methyl carbamate, dimethylol ethyl carbamate and the like. However, with melamine type finishing agents such as trimethylol melamine, methylated trimethylol melamine and the like, the catalysis provided by $Al_2(OH)_5Cl$ is less effective than that provided by magnesium chloride, a relatively mild, common catalyst.

Additives, softeners, modifiers, and other components customarily used in textile finishing pad baths can be used with the catalyst systems of this invention. The composition of the bath is limited only by the compatibility of these ingredients with the catalyst.

The textile materials treated with the catalyst systems of this invention may be in form of fibers, yarns and fabrics. The latter may be woven, knitted, or nonwoven structures. The material may be composed entirely of cellulosic fibers, either natural or regenerated, or may be composed of said cellulosics as components of the textile structure with other cellulosic, noncellulosic natural or synthetic fibers.

Finishing treatments in which the aluminum chlorhydroxide catalyst systems of this invention can be employed include conventional methods such as pad-dry-cure finishing, post-cure finishing, and the like, as well as unusual and specialized finishing treatments such as mild cure finishing, moist cure finishing (damp crosslinking), one-step dry-cure finishing, solvent vapor cure finishing, superheated steam cure finishing, and the like.

Pad-dry-cure finishing is the most common of the treatment methods for producing wrinkle resistant textile fabrics. As the name denotes, the sequence of steps consists of impregnating the textile material by immersing it in the treatment solution (pad bath), squeezing free of excess solution by passing through pad rolls, drying at a moderately elevated temperature (usually so that the temperature of the fabric does not exceed about 100°C), and curing at a higher temperature, generally in the range of 150°–180°C. This type of treatment is sometimes referred to as precure finishing.

Post-cure finishing is a modification of pad-dry-cure finishing in which there is a delay period, often quite extended, between the drying step and the curing step. The fabric, impregnated with finishing agent and catalyst and dried but not cured, is called sensitized fabric. It can be shipped, held in warehouses, and fabricated (cut, sewn, and trimmed) into garments in this state and then creased and shaped prior to the final curing step.

Mild cure finishing is a process for producing wrinkle resistant goods in which cotton or polyester/cotton fabric, impregnated with a solution containing a suitable crosslinking agent and a strong catalyst, is heated at about 60°–100°C for a relatively short period of time without prior drying. During the heat treatment, moisture content of the cotton fibers is reduced to about 3–5 percent and the strong catalyst promotes crosslinking of the cellulose chains with the fibers in a slightly swollen state. This type of treatment gives a finished fabric with a high level of wet wrinkle resistance together with a high level of dry wrinkle resistance. Detailed descriptions of mild cure finishing are given in the Textile Chemist and Colorist, Vol. 1, p. 415, 1969 and Vol. 2, p. 337, 1970.

Moist cure finishing, also called damp crosslinking, is a type of treatment that is popular in Europe for producing fabrics with high wet and dry wrinkle resistance. Cotton or cotton blend fabric is impregnated with a solution of crosslinking agent and strongly acidic catalyst, dried to a specific moisture level in the range of about 6–12 percent, sealed in a package to prevent change of moisture content, held at room temperature for about 12–24 hours, then neutralized and washed. Additional details of this type of finishing are available in U.S. Pat. No. 3,409,462.

One-step dry-cure finishing is a treatment method in which fabric is impregnated with a treatment solution and dried and cured in a one-step operation. These treatments are similar to mild cure finishing (no predry step) except that treatment temperatures ranging up to 180°C are used. With treatment above 100°C, the fabric is essentially completely dry and the fibers are

totally collapsed during the crosslinking reaction. In Germany, treatments of this kind in which drying and curing are carried out in one operation are called "shock curing." Some information on shock curing is given in Textilveredlung, Vol. 5, p. 334, 1970.

A description of solvent vapor cure finishing is available in the Proceedings of AATCC Symposium "Textile Solvent Technology-Update '73," published by the American Association of Textile Chemists and Colorists, p. 79. In this new method of finishing, a solution containing a cellulose crosslinking agent and a catalyst is applied and the impregnated fabric is cured in the vapors from a boiling organic solvent.

Superheated steam cure finishing is a method whereby suitably impregnated fabric is cured in an atmosphere of superheated steam rather than in heated air as in conventional processing.

SUMMARY OF THE INVENTION

The value of wrinkle resistance in textile products is well recognized by those engaged in the textile, garment, retail, and chemical industries as well as by the consumer. It has been estimated that more than 3 billion yards of woven smooth-drying cotton and cotton-blend fabric presently are produced per year in the United States alone. The property of smooth-drying is, of course, a result of the wrinkle resistance of the fabric. To achieve the level of wrinkle resistance needed for smooth-drying, cellulose containing textiles must be chemically treated, usually with formaldehyde or a formaldehyde-amide adduct. The present invention provides catalyst systems for such treatments.

The catalyst systems of the present invention exhibit a number of advantages over catalysts that have been used heretofore. Among these advantages are production of products with improved properties, economies of processing, and the ability to achieve certain treatments under processing conditions previously found impossible. These and other advantages are obvious to those skilled in the art.

Aluminum chlorhydroxide catalyst systems are versatile. They are effective in a diverse range of finishing treatments. In fact, $Al_2(OH)_5Cl$ is unique in that no other known material is available which can function as it does in various catalytic situations. That is, it can serve alone as a catalyst; it can be used with strong mineral and sulfonic acids to moderate their catalytic activity; and it can be used with weaker inorganic acids, organic acids, and certain salts to provide increased catalytic activity through synergistic interaction.

In pad-dry-cure finishing, $Al_2(OH)_5Cl$ functions well as a catalyst. It can be used with a variety of finishing agents, including urea, cyclic urea, uron and carbamate types. $Al_2(OH)_5Cl$ compares favorably with the widely used magnesium chloride, zinc nitrate, and zinc chloride as a catalyst in treatments at 140–160°C. Mixtures of $Al_2(OH)_5Cl$ and these salts are more active as catalysts than either component alone and, thus, permit lower curing temperatures or shorter curing times. For example, chlorine resistant finishes were produced at 120°C whereas curing temperatures 140°–160°C were required when the treatment was catalyzed by the salts or $Al_2(OH)_5Cl$ alone. The savings afforded in processing costs and energy required (particularly in view of current shortages) by use of lower temperatures and shorter times are advantageous features of these catalyst systems.

Effective catalysis in pad-dry-cure finishing at temperatures considerably lower than those of conventional treatments has been obtained with a broad range of compositions consisting of $Al_2(OH)_5Cl$ and various organic acids.

$Al_2(OH)_5Cl$ also is an effective catalyst for post-cure finishing. In treatments with dimethylol dihydroxyethyleneurea, the most commonly employed agent for post-cure processing, little or no reaction takes place in the sensitization treatment of the fabric nor in prolonged storage of the sensitized fabric. Creasing, either with a home-type electric hand iron gives an effective post-cure or with a commercial-type steam press followed by an oven cure produces sharp creases and a smooth pressed appearance in the fabric durable to laundering. Because $Al_2(OH)_5Cl$ has sufficient catalytic activity for post-curing, is relatively inactive in sensitization, and does not have adverse (hydrolytic) effect in unwashed, finished fabrics, it is particularly valuable for this important type of fabric processing.

In mild cure finishing, $Al_2(OH)_5Cl$ alone does not provide sufficient catalysis. When it is used with hydrochloric, nitric, or hydroxymethanesulfonic acid as the catalyst, it functions as a buffer. The pH of the pad bath is raised and the catalytic activity of the strong acid is attenuated. This moderation is manifested in slightly lower wrinkle recovery angles but considerably higher strengths in the finished fabrics. Importantly, the need for neutralization and afterwashing of the finished fabric is precluded. It is well known that without afterwashing, the strength of fabric finished by the mild cure treatment process is further decreased from the hydrolytic action of residual acid. However, if $Al_2(OH)_5Cl$ is included in the treatment, such continued degradation is overcome and strength remains unchanged without an afterwash.

Phosphoric acid which has heretofore been unsuitable for mild cure treatments produced good durable press finishes when used in conjunction with $Al_2(OH)_5Cl$. Synergistic catalyst systems effective in mild cure finishing also resulted from combinations of $Al_2(OH)_5Cl$ and various organic acids. Among these were acetic, lactic, formic, chloroacetic, oxalic, and trichloroacetic acid.

Similar synergism is operative between $Al_2(OH)_5Cl$ and phosphoric acid in moist cure finishing (damp crosslinking). $Al_2(OH)_5Cl$ alone can serve as catalyst for moist cure finishing with urea-formaldehyde. The latter often cannot be used because of polymerization induced by the strong acids usually employed in moist cure finishing.

Catalysis by $Al_2(OH)_5Cl$ is efficient in one-step dry-cure treatments, which are similar to mild cure finishing but at temperatures up to 180°C. Lower treatment temperatures are effective when $Al_2(OH)_5Cl$ is augmented by even as weak an acid as acetic acid. However, crosslinking agents with reactivities as diverse as those of formaldehyde and dimethylol ethyleneurea produce high durable press ratings when catalyzed by $Al_2(OH)_5Cl$ alone.

Greater catalytic activity from a synergistic interaction between $Al_2(OH)_5Cl$ and organic acids is operative also in solvent vapor cure finishing. In this finishing method, the synergism can be obtained in two distinct ways. In one, $Al_2(OH)_5Cl$ and an organic acid are included in the pad bath. The drying and heating action of the vapors from the boiling solvent promote the finishing reaction with the catalyst components

present in the fabric from padding application. In the other embodiment, the finishing agent and $Al_2(OH)_5Cl$ are applied to the fabric by padding which is then cured in the vapors from a boiling mixture of an organic solvent and organic acid. That is, the synergistic catalyst activation results from one catalyst component applied from padding application and the second component from the hot vapors of the boiling solvent-acid mixture.

$Al_2(OH)_5Cl$ catalyst systems are operative with curing in superheated steam similar to their action in heated air. An $Al_2(OH)_5Cl$/lactic acid catalyst system produced higher levels of wrinkle resistance than a magnesium chloride citric acid mixture in fabric finished in superheated steam at 116°C. With steam superheated to 138°C, $Al_2(OH)_5Cl$ alone is sufficiently active as catalyst to give fabric with wrinkle resistance essentially equivalent to that of fabric similarly teeated but in an oven. Strength of the finish produced from curing in steam was about 30% greater than that of the oven-cured finish.

Fully cured durable press fabrics can be creased by techniques that are applicable in commercial plants and in the home when $Al_2(OH)_5Cl$ or $Al_2(OH)_5Cl$ in conjunction with certain mineral acids, organic acids, or salts (as disclosed above) serves as the catalyst system for both the finishing and creasing operations. These catalyst systems permit sharp, high quality, durable creases to be introduced into fully cured durable press fabrics composed of all-cotton and blends of cotton or other cellulosic fibers with synthetic fibers. The resulting creases are sharper and more durable than those which can be introduced into fully cure fabrics that were produced from treatment with conventionally-used catalysts. The term fully cured fabrics is used here in the sense that such fabrics have been treated so that they have a level of durable press smoothness sufficiently high that the fabric can be utilized, albeit without creases, without the need for further processing of any kind.

Creasing of the fully cured fabric can be carried out in several ways. These include: creasing on a commercial steam press followed by oven treatment; creasing on a commercial steam press followed by treatment on a hot-head press; creasing with a household-type hand iron; and creasing with a household steam iron followed by treatment with a hand iron. The first two techniques are applicable for processing in a commercial plant while the last two are suitable for use in the home.

The finished fabric, of course, must not be washed before creasing, as extraction of residual catalyst prevents breaking and reforming of crosslinks in the cotton component and inhibits introduction of a durable crease. Similarly, overcuring in application of the durable press finish must be avoided, or subsequent creasing is poorer.

In addition to the more common $Al_2(OH)_5Cl$, other aluminum chlorhydroxide complexes can be utilized in catalyst systems of the type described above for finishing cellulose containing textile materials for wrinkle resistance. Aluminum chlorhydroxide complexes of the general formula

$$Al_2(OH)_nCl_{6-n}$$

have been used wherein the value of n was 2–5. The acidity of the complex increases as the value of n decreases. The more acidic complexes ($n<5$) afford utilization of even lower concentrations, and less stringent processing conditions, i.e. shorter times and lower temperatures, than those detailed above for $Al_2(OH)_5Cl$ catalyst systems.

DESCRIPTION OF SPECIFIC EMBODIMENTS

The following examples further describe the invention. They are given merely as illustrations and should not be considered as limiting the scope of the invention.

In the examples, percentages reported in compositions and formulations are given as percentages by weight. Temperatures are given in degrees Centigrade. Properties of the fabrics were determined by standard test methods, when available, otherwise, unofficial, but generally recognized methods were used. The following procedures were used: breaking strength by ASTM D1682-64; wrinkle recovery angles, conditioned by AATCC 66-1968 and wet by the same procedure on specimens soaked and blotted; durable press (DP) ratings, tumble-dried (TD) by AATCC 124–1969, 111-B and line-dried (LD) by AATCC 124–1969, 111-A; crease ratings by evaluation under overhead lighting while mounted on an AATCC rating board, scale 5 (best) to 1 (poorest); damage due to retained chlorine (chlorination-scorch test) by AATCC 92-1967.

Nitrogen analyses reported were determined by the Kjeldhal method; formaldehyde analyses, by the chromotropic acid colorimetric method.

EXAMPLE 1

The use of $Al_2(OH)_5Cl$ as catalyst in finishing cotton fabric by the conventional pad-dry-cure method with various formaldehyde-amide adducts is illustrated by the data of Table 1.

TABLE 1

Finishing Agent(1)	Properties of Finished Fabrics					
	N, %	Brk. Str. (W). lb.	Wr. Rec. Ang. (W+F), Cond.	Wr. Rec. Ang. deg. Wet	DP Rating TD	DP Rating LD
Dimethylol ethyleneurea	2.01	24.4	292	277	4.9	4.7
Dimethylol dihydroxyethyleneurea	1.48	21.1	301	282	4.6	4.9
Dimethylol substituted propyleneurea	1.19	31.8	275	245	4.4	4.0
Tetramethylol acetylenediurea	2.07	22.3	291	268	4.9	3.6
Bis(methoxymethyl) uron	1.51	28.5	277	259	4.2	4.4
Dimethylol methyl carbamate	0.90	23.7	282	261	4.5	3.5
Dimethylol ethyl carbamate	0.78	26.6	278	258	4.4	3.7

TABLE 1-continued

Finishing Agent(1)	N, %	Brk. Str. (W), lb.	Wr. Rec. Ang. (W+F), deg. Cond.	Wr. Rec. Ang. (W+F), deg. Wet	DP Rating TD	DP Rating LD
Methylated urea-formaldehyde	2.02	27.7	278	259	4.4	4.3
Untreated	—	48.4	182	166	1.0	1.5

(1)Cotton printcloth impregnated with a solution containing 10% of the indicated finishing agent and 2% $Al_2(OH)_5Cl$, dried for 7 min. at 60°C, cured for 3 min. at 160°C, and washed.

EXAMPLE 2

The use of various concentrations of $Al_2(OH)_5Cl$ as catalyst in the finishing of cotton by the conventional pad-dry-cure method with dimethylol methyl carbamate is illustrated by the data of Table II.

TABLE II

Conc., % $Al_2(OH)_5Cl$(1)	0.25	0.5	0.75	1.0	1.5	2.0
DP Rating (TD)	2.7	3.3	3.5	3.9	4.2	4.5

(1)Cotton printcloth impregnated with a solution containing 10% dimethylol methyl carbamate and the indicated concentration of $Al_2(OH)_5Cl$, dried for 7 min. at 60°C, cured for 3 min. at 160°C, and washed.

EXAMPLE 3

The use of other aluminum chlorhydroxide complexes as catalysts in finishing cotton fabric by the pad-dry-cure method with dimethylol dihydroxyethyleneurea is illustrated by the data of Table III.

TABLE III

Catalyst and Concentration	Treatment(1) Curing Time/Temp. min./deg.C	Wr. Rec. Angle (W+F), Deg. Cond.	Wr. Rec. Angle (W+F), Deg. Wet
2% $Al_2(OH)_{4.67}Cl_{1.33}$	3/100	275	272
1.4% $Al_2(OH)_4Cl_2$	3/100	283	276
1.1% $Al_2(OH)_4Cl_2$	3/120	293	270
1.2% $Al_2(OH)_2Cl_4$	3/120	289	280
2% $Al_2(OH)_{4.67}Cl_{1.33}$	3/160	288	293
1.4% $Al_2(OH)_4Cl_2$	3/160	296	290
Untreated	—	152	172

(1)Cotton printcloth impregnated (about 90% wet pickup) with solution containing 9% dimethylol dihydroxyethyleneurea and the indicated catalyst, dried for 7 min. at 60°C, cured as indicated, and washed.

EXAMPLE 4

A comparison of $Al_2(OH)_5Cl$ and $Zn(NO_3)_2$ as catalysts in the finishing of cotton/polyester blend fabrics by the pad-dry-cure method with dimethylol dihydroxyethyleneurea is illustrated by the data of Table IV.

TABLE IV

Fabric Composition % Cotton	Fabric Composition % Polyester	Treatment(1) Catalyst	Treatment(1) Cure Temp. deg.C	Fabric Properties DP Rating (TD)	Fabric Properties Brk. Str. (W), lb.
75	25	—	—	2.5	135
75	25	$Al_2(OH)_5Cl$	140	4.3	92
75	25	$Zn(NO_3)_2$	160	4.5	85
65	35	—	—	2.5	109
65	35	$Al_2(OH)_5Cl$	140	4.4	100
65	35	$Zn(NO_3)_2$	160	4.4	90
50	50	—	—	3.0	145
50	50	$Al_2(OH)_5Cl$	140	4.7	138
50	50	$Zn(NO_3)_2$	160	4.7	129
35	65	—	—	3.3	160
35	65	$Al_2(OH)_5Cl$	140	4.6	148
35	65	$Zn(NO_3)_2$	160	4.5	122

(1)Cotton polyester twills impregnated with solution containing 9% dimethylol dihydroxyethyleneurea and 2% $Al_2(OH)_5Cl$ or 0.6% $Zn(NO_3)_2 \cdot 6H_2O$, dried for 7 min. at 60°C, cured for 3 min. at the indicated temperature, and washed.

EXAMPLE 5

The use of $Al_2(OH)_5Cl$/lactic acid catalyst systems in finishing cotton fabric by the pad-dry-cure method with dimethylol dihydroxyethyleneurea is illustrated by the data of Table 5. The synergistic interaction of the catalyst components and the unusually low (for pad-dry-cure finishing) curing temperature should be especially noted.

TABLE V

Catalyst concentration(1)							
% $Al_2(OH)_5Cl$	2.0	1.67	1.33	1.0	0.67	0.33	0.0
% lactic acid	0.0	0.33	0.67	1.0	1.33	1.67	2.0
DP Rating (TD)	3.3	4.3	4.5	4.9	4.7	4.4	1.6

(1)Cotton printcloth impregnated with a solution containing 9% dimethylol dihydroxyethyleneurea and the indicated concentration of $Al_2(OH)_5Cl$ and lactic acid, dried for 7 min. at 60°C, cured for 3 min. at 110°C, and washed.

EXAMPLE 6

The use of $Al_2(OH)_5Cl$, $Al_2(OH)_5Cl/Zn(NO_3)_2$, and $Al_2(OH)_5Cl/ZnCl_2$ catalyst systems in finishing cotton fabric by the pad-dry-cure method at various curing temperatures with dimethylol dihydroxyethyleneurea is illustrated by the data of Table VI. Treatments in which $Zn(NO_3)_2$ and $ZnCl_2$ alone served as catalysts are included for comparison.

TABLE VI

Treatment(1) Catalyst System $Zn(NO_3)_2 \cdot 6H_2O$ %	$ZnCl_2$ %	$Al_2(OH)_5Cl$ %	Curing Temp., deg. C	Properties of Finished Fabrics N, %	Brk. Str. (W), lb.	Str. Retained in Cl-Scorch Test, %	DP Rating TD	DP Rating LD
0.5	—	—	100	0.88	39.1	7	2.6	2.7
0.5	—	—	120	1.31	33.6	16	3.7	3.5
0.5	—	—	140	1.41	30.1	88	4.6	4.0
0.5	—	—	160	1.41	27.4	94	4.7	4.5
0.5	—	2	100	1.12	33.6	42	3.3	3.3
0.5	—	2	120	1.29	30.1	94	4.0	4.2
0.5	—	2	140	1.36	24.0	100	4.7	3.7
0.5	—	2	160	1.34	22.3	96	4.9	4.2

TABLE VI-continued

Treatment(1) Catalyst System $Zn(NO_3)_2 \cdot 6H_2O$ %	$ZnCl_2$ %	$Al_2(OH)_5Cl$ %	Curing Temp., deg. C	N, %	Properties of Finished Fabrics Brk. Str. (W), lb.	Str. Retained in Cl-Scorch Test, %	DP Rating TD	LD
—	—	2	100	0.95	36.1	26	2.7	2.9
—	—	2	120	1.39	31.9	58	4.0	3.5
—	—	2	140	1.34	26.3	99	4.7	3.6
—	—	2	160	1.31	24.0	92	4.9	4.6
—	1	2	100	1.10	31.6	31	3.7	3.0
—	1	2	120	1.25	26.1	90	4.6	3.3
—	1	2	140	1.27	23.0	97	4.7	4.5
—	1	2	160	1.09	20.5	94	4.6	4.5
—	1	—	100	0.53	44.0	7	2.5	2.3
—	1	—	120	1.06	33.4	11	3.3	2.9
—	1	—	140	1.30	28.6	34	4.3	3.3
—	1	—	160	1.37	27.4	90	4.4	4.3
	(Untreated)		—	—	49.6	—	1.0	1.5

(1)Cotton printcloth impregnated with a solution containing 9% dimethylol dihydroxyethyleneurea and the indicated catalyst system, dried for 7 min. at 60°C, cured for 3 min. at the temperature listed (100–160°C), and washed.

EXAMPLE 7

The use of $Al_2(OH)_5Cl/MgCl_2$ catalyst system, and a comparison with $MgCl_2$ and $Al_2(OH)_5Cl$ used alone as catalysts, in finishing cotton fabric by the pad-dry-cure method at various temperatures with dimethylol dihydroxyethyleneurea is illustrated in Table VII.

TABLE VII

Treatment(1) Catalyst System $Al_2(OH)_5Cl$ %	$MgCl_2 \cdot 6H_2O$ %	Curing Temperature deg. C.	DP Rating (TD)
2	—	80	1.5
2	—	100	2.7
2	—	120	4.0
2	—	140	4.7
2	—	160	4.9
—	2.5	80	1.0
—	2.5	100	2.3
—	2.5	120	3.3
—	2.5	140	3.7
—	2.5	160	4.2
2	2.5	80	1.9
2	2.5	100	3.0
2	2.5	120	4.2
2	2.5	140	4.3
2	2.5	160	4.5

(1)Cotton printcloth impregnated with a solution containing 9% dimethylol dihydroxyethyleneurea and the indicated catalyst system, dried for 7 min. at 60°C, cured for 3 min. at the temperature listed (80–160°C), and washed.

EXAMPLE 8

The use of various $Al_2(OH)_5Cl$/organic acid catalyst systems in finishing cotton fabric by the pad-dry-cure method with dimethylol dihydroxyethyleneurea is illustrated in Table VIII.

TABLE VIII

Catalyst System,(1) conc., mmol./100 g pad bath $Al_2(OH)_5Cl$	Organic acid	Properties of Finished Fabrics %N	Brk. Str. lb.	Wr. Rec. Ang. (W+F), deg. Cond.	Wet	DP Rating TD	LD
5	—	0.72	34.8	249	223	2.9	2.6
2.5	2.5 propionic	0.62	37.9	230	215	3.2	2.9
—	5 propionic	0.05	50.6	183	155	1.3	1.5
2.5	2.5 succinic	0.83	30.4	267	249	4.0	3.4
—	5 succinic	0.22	52.4	174	191	1.2	2.0
2.5	2.5 malic	0.93	27.0	280	257	4.4	3.7
—	5 malic	0.45	46.0	213	191	1.7	2.4
2.5	2.5 citric	0.88	19.8	285	266	4.7	3.5
—	5 citric	0.60	36.2	217	216	2.3	2.5
3	3 acetic	0.76	30.4	—	—	3.2	2.6
3	3 glycolic	0.82	27.3	—	—	4.3	4.5
3	3 cyanoacetic	0.87	23.6	—	—	4.3	3.7
3	3 lactic	0.83	27.4	—	—	3.7	3.2
3	3 acrylic	0.73	29.9	—	—	3.5	3.5
3	—	0.57	37.1	—	—	2.0	2.0

(1)Cotton printcloth impregnated with a solution containing 9% dimethylol dihydroxyethyleneurea and the indicated catalyst system, dried for 7 min. at 60°C, cured for 3 min. at 120°C, and washed.

EXAMPLE 9

A sample of cotton printcloth was impregnated with a solution containing 10 percent dimethylol ethyleneurea and 2 percent $Al_2(OH)_5Cl$, dried for 7 min. at 60°C, and cured for 3 min. at 160°C. The sample was divided into five portions. One portion was washed immediately after curing. The remaining portions were stored under ambient room conditions and a portion withdrawn periodically and washed until the final portion had been held for 70 days prior to washing. The various portions were then subjected to the chlorination-scorch test (AATCC Test Method 92-1967) to determine the damage due to retained chlorine. Wrinkle resistance also was determined on the first and last portion. The results are shown in Table IX.

TABLE IX

Days held before washing	Str. Retained in Cl-Scorch Test, %	Wr. Rec. Angle (W+F), deg. Cond.	Wet
0	100	269	248
14	97	—	—
28	102	—	—
56	84	—	—

TABLE IX-continued

Days held before washing	Str. Retained in Cl-Scorch Test, %	Wr. Rec. Angle (W+F), deg. Cond.	Wet
70	82	267	251

These data were compared with those given in FIG. 2, American Dyestuff Reporter, vol. 49, p. 531, 1960 for similar treatments but with zinc nitrate, magnesium chloride, and amine hydrochloride catalysts. Resistance to chlorine damage was rapidly lost by the fabrics treated with zinc nitrate, magnesium chloride, and amine hydrochloride catalysis, i.e., more than 50% strength was lost in tests carried out on these samples after only 21 days storage in the unwashed state. However, the greater stability of the dimethylol ethyleneurea-finished cotton in the presence of residual $Al_2(OH)_5Cl$ catalyst is evident from Table IX, above; even after 70 days storage, strength retained in the chlorination/scorch test was more than 80 percent. Furthermore, the wrinkle resistance of the latter was unaffected by the storage.

EXAMPLE 10

The use of $Al_2(OH)_5Cl$ as catalyst in post cure finishing of cotton-containing fabrics is illustrated in Table X. Essentially equivalent results were obtained in similar treatments of cotton printcloth.

TABLE XI

Catalyst System(1) HCl %	$Al_2(OH)_5Cl$ %	N %	Properties of Finished Fabric Brk. Str. (W) lb.	Wr. Rec. Ang. (W+F), deg. Cond.	Wet
Untreated		—	50.2	179	155
	Cure: 7 min. at 60°C				
—	2	0.06	50.1	174	167
0.37	—	1.21	27.6	274	280
0.37	2	0.86	38.7	223	216
	Cure: 2 min. at 100°C				
—	2	0.43	50.4	179	175
0.37	—	1.25	23.1	271	287
0.37	1.5	1.20	28.1	262	272
0.37	2	1.20	28.6	261	266
0.37	3	1.19	30.6	264	270
0.19	—	1.18	22.6	282	279
0.37	—	1.26	22.6	285	298
0.56	—	1.31	21.1	285	295
0.74	—	1.23	21.1	286	304
1.11	—	1.29	19.1	276	308
0.19	2.5	1.07	29.1	272	265
0.37(2)	2.5	1.10	29.6	269	283
0.56	2.5	1.11	28.6	274	286
0.74	2.5	1.14	28.6	278	293
1.11	2.5	1.20	27.6	273	300

(1)Cotton printcloth impregnated with a solution containing 15% dimethylol methyl carbamate and the indicated catalyst system, cured under the conditions shown (no predry), and washed.

(2)A portion of this fabric was stored without washing (after curing). Strength remained unchanged through 84 days of storage without an afterwash. This is in contrast to the loss suffered upon storage in the unwashed state by fabrics treated similarly but without $Al_2(OH)_5Cl$ in the catalyst system, c.f. FIG. 4 in Textile Chemist & Colorist, Vol. 1, p. 417, 1969.

EXAMPLE 12

TABLE X

Treatment(1)	%N	Brk. Str. % of Untr.	Wr. Rec. Angle, (W+F), deg. Cond.	Wet	DP Rating TD	LD	Crease Rating (TD)
		50/50 cotton/polyester poplin					
Sensitized							
Stored 0 days and washed	0.03	99	231	247	3.0	2.6	—
Stored 90 days and washed	0.32	95	240	266	3.3	3.2	—
Sensitized, Steam pressed, and Cured							
Stored 0 days, pressed, cured, and washed	0.91	86	274	277	4.6	2.7	5
Stored 90 days, pressed, cured, and washed	0.84	83	281	278	4.5	3.5	5
		50/50 cotton/polyester twill					
Sensitized							
Stored 0 days and washed	0.04	100	225	232	3.2	3.5	—
Stored 90 days and washed	0.44	85	231	248	3.3	3.4	—
Sensitized, Steam Pressed, and Cured							
Stored 0 days, pressed, cured, and washed	0.82	68	263	260	4.9	3.7	5
Stored 90 days, pressed, cured, and washed	0.85	69	264	262	4.6	3.7	5

(1)Fabrics sensitized by impregnating with a solution containing 9% DMDHEU and 2% $Al_2(OH)_5Cl$, and drying at 60°C. Storage was at ambient room conditions. Swatches were creased on a commercial-type steam press and cured in an oven at 160°C; poplin for 3 min., twill for 4 min.

EXAMPLE 11

The use of $Al_2(OH)_5Cl$/HCl catalyst systems in mild cure finishing of cotton fabric with dimethylol methyl carbamate is illustrated in Table XI.

The use of $Al_2(OH)_5Cl$/phosphoric acid catalyst systems in mild cure finishing of cotton fabric with dimethylol methyl carbamate is illustrated in Table XII.

TABLE XII

Catalyst System(1) H_3PO_4 %	$Al_2(OH)_5Cl$ %	N %	Properties of Finished Fabric Brk. Str. (W) lb.	Wr. Rec. Ang. (W+F) deg. Cond.	Wet	DP Rating TD	LD
Untreated		—	48.8	196	153	—	
—	2	0.43	50.4	179	175	1.3	1.8
1.5	—	0.94	39.5	215	193	3.2	2.2
1.5	1	1.13	23.9	273	255	3.7	2.7
1.5	2	1.11	28.3	277	242	4.0	3.0
1.5	4	1.08	31.2	266	236	4.3	3.3
0.5	2	0.92	39.5	259	236	3.7	2.9
1	2	1.01	31.7	262	250	4.0	3.4
2	2	1.12	26.8	284	281	4.7	3.9

TABLE XII-continued

Catalyst System(1) H_3PO_4 %	$Al_2(OH)_5Cl$ %	N %	Properties of Finished Fabric Brk. Str. (W) lb.	Wr. Rec. Ang. (W+F) Cond.	deg. Wet	DP Rating TD	LD
3	2	1.12	27.8	274	267	4.7	4.0
2	1	1.20	29.8	279	268	4.7	4.2
2	3	1.08	33.7	279	258	4.4	3.6

(1)Cotton print cloth impregnated with solutions containing 15% dimethylol methyl carbamate and the indicated catalyst system, cured for 2 min. at 100° C. (no predry), and washed.

EXAMPLE 13

The use of $Al_2(OH)_5Cl$/organic acid catalyst systems in mild cure finishing of cotton is illustrated in Table XIII.

TABLE XIII

Acid**	15% DMMC + Acid* N, %	Brk. Str. (W), lb.	Wr. Rec. Ang. (W+F), deg. Cond.	Wet	DP Rating TD	LD	15% DMMC + Acid + 2% $Al_2(OH)_5Cl$* N, %	Brk. Str. (W), lb	Wr. Rec. Ang (W+F), deg. Cond	Wet	DP Rating TD	LD
1.2% Acetic Acid	0.02	48.8	172	171	1.2	2.0	0.68	37.5	237	220	3.0	2.4
1.8% Lactic Acid	0.07	49.3	176	156	1.5	2.0	0.97	36.0	258	247	3.5	3.3
0.92% Formic Acid	0.03	49.3	174	163	1.2	2.0	0.85	37.0	252	229	3.3	3.3
1.89% Chloro-acetic Acid	0.15	49.3	165	165	1.2	2.0	1.13	30.6	275	267	3.9	3.9
2.52% Oxalic Acid	0.99	31.6	236	219	2.7	2.7	1.10	30.6	272	277	4.0	3.5
3.27% Trichloro-acetic Acid	1.20	33.5	255	244	3.3	3.6	1.21	30.6	268	286	4.0	4.4
None	—	—	—	—	—	—	0.32	48.8	186	179	1.6	2.0

*Cotton print cloth impregnated with the indicated solution (DMMC = dimethylol methyl carbamate), cured for 2 min. at 100°C. (no predry), and washed. Breaking strength of the untreated fabric was 49.3 lb.; wrinkle recovery angles were 189° (cond.) and 133° (wet).
**The concentrations employed were equivalent to 20 mmol. of acid per 100 g of solution.

EXAMPLE 14

The use of $Al_2(OH)_5Cl$/phosphoric acid and $Al_2(OH)_5Cl$/lactic acid catalyst systems in moist cure finishing (damp crosslinking) of cotton fabric with dimethylol methyl carbamate is illustrated by the data of Table XIV.

TABLE XIV

Treatment* Catalyst System H_3PO_4, %	$Al_2(OH)_5Cl$, %	Lactic Acid, %	pH of bath	Properties of Finished Fabric N, %	Brk. Str. (W), lb.	DP Rating TD	LD
1.96	—	—	1.85	0.61	46.3	2.5	2.3
1.96	2	—	2.0	0.93	36.5	3.9	3.3
—	2	—	4.2	0.08	51.0	1.4	1.7
—	—	1.8	2.4	0.05	49.2	1.5	1.7
—	2	1.8	2.8	0.62	44.3	2.7	2.3
	(Untreated)		—	—	49.6	1.0	1.6

*Cotton print cloth impregnated with a solution containing 15% dimethylol methyl carbamate and the indicated catalyst system, dried at 60°C. to about 6% moisture, sealed in plastic bags and held for 24 hours at room temperature, and washed.

The use of urea-formaldehyde (UF) in moist cure finishing is very difficult; in many cases, impossible. Precipitation of the pad bath due to polymerization of the UF occurs immediately upon addition of the strong acids usually used as catalysts. However, 2% $Al_2(OH)_5Cl$ as catalyst with UF in moist cure treatment (as detailed in the footnote to Table XIV) gave a finished cotton print cloth with a DP rating (TD) of 3.6 and a breaking strength equal to 69% of the untreated cotton. The pH of this bath was 4.1; there was no trouble with polymerization of the pad bath.

EXAMPLE 15

The use of $Al_2(OH)_5Cl$ and $Al_2(OH)_5Cl$/acetic acid catalyst systems in one-step dry-cure finishing of cotton fabric with formaldehyde is illustrated by the data of Table XV.

TABLE XV

Treatment* Temp. deg. C.	10% HCHO, 1.2% Acetic Acid, 2% $Al_2(OH)_5Cl$ HCHO, %	DP Rating TD	10% HCHO, 2% $Al_2(OH)_5Cl$ HCHO, %	DP Rating TD
100	0.22	2.6	0.03	1.5
120	1.46	3.7	0.40	2.9
140	2.22	4.5	1.15	4.0
160	2.51	4.5	1.81	4.3
180	2.27	4.5	2.02	4.4

*Print cloth impregnated with the indicated solution, cured for 2 min. at 100–180° C., (no predry) and washed. Durable press rating of the untreated fabric was 1.0.

EXAMPLE 16

The use of $Al_2(OH)_5Cl$ and $Al_2(OH)_5Cl$/acetic acid catalyst systems in one-step dry-cure finishing of cotton with dimethylol methyl carbamate is illustrated by the data of Table XVI.

TABLE XVI

Treatment* Catalyst System				Properties of Finished Fabric		
Acetic Acid %	$Al_2(OH)_5Cl$ %	Temp., Deg. C	N, %	Brk. Str. (W), lb.	DP Rating TD	DP Rating LD
1.2	2	80	0.04	47.9	1.6	2.2
1.2	2	100	0.85	32.1	3.3	2.6
1.2	2	120	1.15	29.1	4.4	3.3
1.2	2	140	1.20	25.0	4.5	3.7
1.2	2	160	1.15	23.3	4.7	4.3
1.2	2	180	1.14	22.2	4.6	4.7
1.2	—	100	0.02	49.6	1.0	1.8
1.2	—	160	0.11	47.7	1.4	1.9
—	2	80	0.04	53.2	1.5	1.7
—	2	100	0.36	47.6	2.3	2.0
—	2	120	0.72	37.8	2.9	2.6
—	2	140	0.98	34.4	3.7	2.9
—	2	160	1.08	30.1	4.0	3.7
—	2	180	1.10	26.0	4.2	3.7
(Untreated)			—	50.5	1.3	2.0

*Cotton print cloth impregnated with a solution containing 15% dimethylol methyl carbamate and the indicated catalyst system, (no predry) cured for 2 min. at the indicated temperature, and washed.

EXAMPLE 17

The use of aluminum chlorhydroxides in the catalysis of finishing treatments with curing in the vapors from boiling solvents rather than in an atmosphere of heated air is illustrated by the data of Table XVII. Curing in the vapors from boiling perchloroethylene and from a 98:2 mixture (by weight) of perchloroethylene and acetic acid are exemplified in this table.

TABLE XVII

Treatment		Properties of Finished Fabric			
				Br. Rec. Angle (W+F). deg.	
Pad Bath*	Solvent Vapor**	%N	Brk. Str. % of Untr.	Cond.	Wet
DMDHEU	Perchl./acetic	0.27	98	190	209
DMDHEU/$Al_2(OH)_5Cl$	Perchl./acetic	0.73	59	249	269
DMDHEU/$Al_2(OH)_5Cl$	Perchloroethylene	0.60	79	234	220
DMDHEU/$Al_2(OH)_4Cl_2$	Perchloroethylene***	1.43	46	292	291
Untreated	—	—	100 (50.6 lb.)	206	162

* Cotton print cloth impregnated (about 90% wet pickup) with solutions containing 9% DMDHEU, 9% DMDHEU/2% $Al_2(OH)_5Cl$, or 9% DMDHEU/1.1% $Al_2(OH)_4Cl_2$ as indicated. DMDHEU = dimethylol dihydroxyethyleneurea.

** Fabric dried for 7 min. at 60°C, cured in the vapors of boiling solvent for 3 min., and washed. Vapors from perchloroethylene were 121°C, and those from 98:2 mixture of perchloroethylene and acetic acid were 106°C.

***Cured for 1 min.

EXAMPLE 18

The use of $Al_2(OH)_5Cl$ catalyst systems in finishing treatments with curing carried out in superheated steam rather than in an atmosphere of heated air is illustrated by the data of Table XVIII.

TABLE XVIII

Treatment*		Properties of Finished Fabric		
		Brk. Str.	Wr.Rec.Ang.(W+F), dg.	
Catalyst System	%N	(W), lb.	Cond.	Wet
Pad, Dry,Steam-Cure (3 min./116°C)				
2% $Al_2(OH)_5Cl$	0.72	42.5	233	202
1% $Al_2(OH)_5Cl$/1% lactic acid	0.89	34.2	268	255
Pad,No predry,Steam-Cure (6 min./116°C)				
2% $Al_2(OH)_5Cl$	0.60	47.8	210	206
1% $Al_2(OH)_5Cl$/1% lactic acid	0.84	37.4	264	253
Pad.Dry,Steam-Cure (3 min./138°C)				
2% $Al_2(OH)_5Cl$	0.90	34.9	264	234
Pad, No predry, Steam-Cure (6 min./138°C)				
2% $Al_2(OH)_5Cl$	0.96	37.6	268	240
Untreated	—	49.4	178	170

*Cotton print cloth impregnated (90% wet pickup) with solutions containing 9% dimethylol dihydroxyethyleneurea and the indicated catalyst system, dried for 7 min. at 60°C or not predried as indicated, cured in a chamber in an atmosphere of superheated steam for the time and temperature listed, and washed.

EXAMPLE 19

A comparison showing the superiority of fully cured fabrics that have been finished with $Al_2(OH)_5Cl$ catalyst for subsequent creasing is given in Table XIX.

TABLE XIX

		Crease Rating			
	Cotton. %	35	50	65	75
Fully Cured Twill(a)	Polyester. %	65	50	35	25
DMDHEU/$Al_2(OH)_5Cl$					
Steam Press - Oven(b)		5	5	5	3.5
Steam Press - Not Head Press(c)		5	5	4.2	2
Hand Iron(d)		5	5	5	4.6
Steam Iron - Hand Iron(e)		5	5	5	2.3
DMDHEU/$Zn(NO_3)_2$					
Steam Press - Oven(b)		3.3	2.5	1.5	2.5
Steam Press - Hot Head Press(c)		2.3	3.9	2.7	1.5
Hand Iron(d)		5	4.3	3	3
Steam Iron - Hand Iron(e)		4.5	4.2	2.6	3

(a)Cotton/polyester twills (of various fiber compositions) were fully cured by pad-dry-cure treatments with 9% dimethylol dihydroxyethyleneurea (DMDHEU) and 2% $Al_2(OH)_5Cl$, and with 9% DMDHEU and 0.6% $Zn(NO_3)_2 \cdot 6H_2O$. They were not washed after curing. The fully cured fabrics were creased, as shown, than washed and tumble dried. DP ratings of the fully cured fabrics were 4.3–4.6. These values were essentially unchanged by the subsequent creasing treatments.

(b)Creased by pressing on a commercial-type steam press at about 120°C. for 15

TABLE XIX-continued

		Crease Rating			
	Cotton. %	35	50	65	75
Fully Cured Twill(a)	Polyester. %	65	50	35	25

sec. turned over, and pressed again. The creased fabric was then placed in a circulating air oven at 160°C. for 3 min.
(c)Creased, as above on the steam press, then the creased fabric was heat treated on a hot head press at 120°C. for 30 sec., turned over, and pressed again.
(d)Creased by pressing with a home-type hand iron, surface temperature 180°C., for 1 min.
(e)Creased by pressing with a home-type steam iron, then pressed with a home-type hand iron, surface temperature 165°C. for 1 min.

EXAMPLE 20

The creasing of fully cured all-cotton and cotton/polypropylene poplin fabrics that had been finished in $Al_2(OH)_5Cl$ and $Al_2(OH)_5Cl$/lactic acid catalysis is illustrated in Table XX.

TABLE XX

	Finishing Treatment	
	9% DMDHEU	9% DMDHEU
	9% DMDHEU	2% $Al_2(OH)_5Cl$
Property	2% $Al_2(OH)_5Cl$	0.2% lactic acid

We claim:

1. In a method of preparing wrinkle resistant, cellulosecontaining textile wherein said textile is (a) impregnated with a solution consisting essentially of water, a catalyst and a finishing agent selected from the group consisting of formaldehyde and formaldehyde-amide adducts, (b) dried and cured, and (c) washed, the improvement comprising the use of aluminum chlorhydroxide complexes of the formula $Al_2(OH)_nCl_{6-n}$, wherein n has a value of 2-5, as said catalyst.

2. A process for preparing cellulose-containing textiles with improved wrinkle resistance which process comprises:
   a. padding a cellulose-containing fabric in an aqueous solution consisting essentially of water, about 0.1 to 5 weight percent of an aluminum chlorhydroxide complex of the formula $Al_2(OH)_nCl_{6-n}$ wherein n has a value of 2–5 and about 9–15 weight percent of a finishing agent selected from the group consisting of formaldehyde and formaldehyde-amide adducts,
   b. drying the fabric from (a) at a temperature of about 60°C. for a period of about 7 minutes, and
   c. curing the fabric from (b) at a temperature of about 100°–160°C. for a period of about 3 minutes.

3. A process for preparing cellulose-containing textiles with improved wrinkle resistance which process comprises:
   a. padding a cellulose-containing fabric in on aqueous solution containing about 0.1 to 5 weight percent of an aluminum chlorhydroxide complex of the formula $Al_2(OH)_nCl_{6-n}$ wherein n has a value of 20-5, about 9 to 15 weight percent of a finishing agent selected from the group consisting of formaldehyde and formaldehyde-amide adducts, and about 0.1 to 5 weight percent of an organic acid,
   b. drying the fabric from (a) at a temperature of about 60°C. for a period of about 7 minutes, and
   c. curing the fabric from (b) at a temperature of about 100°–160°C. for a period of about 3 minutes.

4. A process for preparing cellulose-containing textiles with improved wrinkle resistance which process comprises:
   a. padding a cellulose-containing fabric in an aqueous solution containing about 2 weight percent of an aluminum chlorhydroxide complex of the formula $Al_2(OH)_nCl_{6-n}$ wherein n has a value of 2-5, about 9–15 weight percent of a finishing agent selected from the group consisting of formaldehyde and formaldehydeamide adducts and about 0.2 to 3 weight percent of a salt selected from the group consisting of zinc chloride, zinc nitrate and magnesium chloride,
   b. drying the fabric from (a) at a temperature of about 60°C for a period of about 7 minutes, and
   c. curing the fabric from (b) at a temperature of about 100°–160°C. for a period of about 3 minutes.

5. A process for preparing cellulose-containing textiles with improved wrinkle resistance which process comprises:
   a. padding a cellulose-containing fabric in an aqueous solution containing about 0.1 to 5 weight percent of an aluminum chlorhydroxide complex of the formula $Al_2(OH)_nCl_{6-n}$ wherein n has a value of 2-5, about 9 to 15 weight percent of a finishing agent selected from the group consisting of formaldehyde and formaldehyde-amide adducts, and about 0.1 to 5 weight percent of an acid selected from the group consisting of hydrochloric, nitric, hydroxymethanesulfonic, phosphoric acid, and
   b. reducing the moisture content of the fabric from (a) to about 3-5 percent by curing said fabric by heating at a temperature of about 60°–100°C. for a period of about 2 to 7 minutes, the higher temperature requiring the lesser time.

6. A process for preparing cellulose-containing textiles with improved wrinkle resistance which process comprises:
   a. padding a cellulose-containing fabric with an aqueous solution containing about 0.1 to about 5 weight percent of an aluminum chlorhydroxide complex of the formula $Al_2(OH)_nCl_{6-n}$ wherein n has a value of 2–5, about 9 to 15 weight percent of a finishing agent selected from the group consisting of formaldehyde and formaldehyde-amide adducts, and about 0.1 to 5 weight percent of an organic acid, and
   b. reducing the moisture content of the fabric from (a) to about 3-5 percent by heating at a temperature of about 60°–100°C. for a period of about 2 to 7 minutes, the higher temperature requiring the lesser time.

7. A process for preparing cellulose-containing textiles with improved wrinkle resistance which process comprises:
   a. padding a cellulose-containing fabric with an aqueous solution containing about 15 weight percent of dimethylol methyl carbamate, about 2 weight percent of an aluminum chlorhydroxide complex of the formula $Al_2(OH)_nCl_{6-n}$ wherein n has a value of 2–5, and about 0-2 weight percent of an acid selected from the group consisting of phosphoric and lactic acid, and
   b. drying the fabric from (a) at a temperature of about 60°C. to a moisture content of about 6 percent, and
   c. curing the fabric from (b) at room temperature for about 24 hours, with the fabric sealed in a package to prevent change of moisture content.

8. A process for preparing cellulose-containing textiles with improved wrinkle resistance which process comprises:

a. padding a cellulose-containing fabric with an aqueous solution consisting essentially of water, about 15 weight percent of urea-formaldehyde and about 2 weight percent of an aluminum chlorhydroxide complex of the formula $Al_2(OH)_5Cl$, and

b. drying the fabric from (a) at a temperature of about 60°C. to a moisture content of about 6 percent, and

c. curing the fabric from (b) at room temperature for about 24 hours, with the fabric sealed in a package to prevent change of moisture content.

9. A process for preparing cellulose-containing textiles with improved wrinkle resistance which process comprises:

a. padding a cellulose-containing fabric with an aqueous solution containing about 9 to 15 weight percent of a finishing agent selected from the group consisting of formaldehyde and formaldehyde-amide adducts, about 0.1 to 5 weight percent of an aluminum chlorhydroxide complex of the formula $Al_2(OH)_nCl_{6-n}$ wherein n has a value of 2-5, and from 0 to 5 weight percent of an organic acid, and

b. curing the fabric from (a) by heating at a temperature of about 80° to 180°C. for a period of about 2 minutes.

10. A process for preparing cellulose-containing textiles with improved wrinkle resistance which process comprises:

a. padding a cellulose-containing fabric with an aqueous solution consisting essentially of water, about 9 to 15 weight percent of a finishing agent selected from the group consisting of formaldehyde and formaldehyde-amide adducts, and about 0.1 to 5 weight percent of an aluminum chlorhydroxide complex of the formula $Al_2(OH)_nCl_{6-11n}$ wherein n has a value of 2-5, and

b. drying the fabric from (a) at a temperature of about 60°C. for a period of about 7 minutes, and

c. curing the fabric from (b) for from about 1 to 3 minutes in the vapors from a boiling solution selected from the group consisting of perchloroethylene and a mixture of 98 weight percent perchloroethylene and 2 weight percent acetic acid.

11. A process for preparing cellulose-containing textiles with improved wrinkle resistance which process comprises:

a. padding a cellulose-containing fabric with an aqueous solution-containing about 9 weight percent of dimethylol dihydroxyethyleneurea, about 1 to 2 weight percent of an aluminum chlorhydroxide complex of the formula $Al_2(OH)_5Cl$, and about 0–1 weight percent of lactic acid, and,

b. drying the fabric from (a) at a temperature of about 60°C for 7 minutes, and

c. curing the fabric from (b) by treatment with superheated steam for about 3 minutes at a temperature of about 116°–138°C.

12. The process of claim 11 wherein the fabric from (a) is cured by treatment with superheated steam for about 6 minutes at a temperature of about 116°–138°C.

13. A process for preparing durably creased, wrinkle resistant cellulose-containing textiles which process comprises:

a. padding a cellulose-containing fabric with an aqueous solution consisting essentially of water, about 9 weight percent of dimethylol dihydroxyethyleneurea and about 2 weight percent of an aluminum chlorhydroxide complex of the formula $Al_2(OH)_nCl_{6-n}$ wherein n has a value of 2-5, and

b. drying the fabric from (a) at about 60°C., and c. creasing the fabric from (b) by steam pressing, and d. curing the fabric from (c) by heating in an oven at about 160°C. for a period of from 3 to 4 minutes.

14. A process for preparing durably creased, wrinkle resistant cellulose-containing textiles which process comprises:

a. padding a cellulose-containing fabric with an aqueous solution containing about 9 to 15 weight percent of a finishing agent selected from the group consisting of formaldehyde and formaldehyde-amide adducts, about 0.1 to 5 weight percent of an aluminum chlorhydroxide complex of the formula $Al_2(OH)_nCl_{6-n}$ where n has a value of 2–5, and about 0 to 5 weight percent of an organic acid, and

b. drying the fabric from (a) at a temperature of about 60°C. for a period of about 7 minutes and

c. curing the fabric from (b) at a temperature of about 80° to 180°C. for a period of from about 2 to 3 minutes, and,

d. creasing the fully cured fabric from (c) by a method selected from the group consisting of

i. creasing by pressing on a commercial-type steam press at about 120°C. for 15 seconds, turning the creased fabric over and pressing again, followed by heating in an oven at about 160°C. for about 3 minutes.

ii. creasing on a steam press as in (i) then pressing on a hot head press at 120°C for 30 seconds, turning the creased fabric over, and pressing again.

iii. creasing by pressing with a home-type hand iron with surface temperature at 180°C. for about 1 minute.

iv. creasing by pressing with a home-type steam iron, followed by pressing with a home-type hand iron with surface temperature at 165°C. for about 1 minute.

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