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PROCESS FOR THE PRODUCTION OF CELLULOSIC TEXTILES WITH PERMANENT CREASES AND IMPROVED SOIL AND ABRASION RESISTANCE

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A non-exclusive, irrevocable, royalty-free license in the invention herein described, throughout the world for all purposes of the United States Government, with the power to grant sublicenses for such purposes, is hereby granted to the Government of the United States of America.

This invention relates to a process of treating a cellulosic textile material such as a natural cellulosic textile material like cotton, ramie, and linen, and including also synthetic cellulosic fibers like rayon, to produce a finish thereon which will cause the material to retain permanent creases, exhibit excellent wrinkle-resistance, dimensional stability, shape holding quality and resistance to soiling.

It has heretofore been possible to impart permanent creases and/or wrinkle resistance to cellulosic textile materials through the application of thermosetting, nitrogen-containing, methylol resins in conjunction with soft, thermoplastic polymers such as the acrylates with or without polymeric silicone lubricants. The permanent crease imparting finish thus introduced is permanent to at least 20 laundering cycles. Cotton garments treated by these conventional processes possess wrinkle resistance and dimensional stability (shape holding qualities). A serious disadvantage exists, however, in the aforementioned processes, in that these conventional finishes are readily susceptible to soiling and will, in fact, scavenge dirt from the wash water during a laundering process. In the instant invention, the production of the finish having the desirable properties described above are achieved by the elimination of the soft, thermoplastic polymers and the polymeric silicone lubricants while still retaining the lubricating effect of the latter for ease of padding or curing.

In accordance with the invention, a cellulosic textile material, such as cotton, linen, ramie, and rayon, and which may be in the form of fiber, yarn, fabric, or an intermediate form thereof, is impregnated, as, for example, by means of pad rolls, mangles, quetches, centrifugals, or hydroextractors, with a water emulsion containing, based on the solids content, about from 1 to 15 weight percent of dimethylol cyclic ethylene urea, about from 0.1 to 5 weight percent of polyethylene (as a thermoplast), preferably emulsified polyethylene and about from 0.1 to 5 weight percent of a latent curing catalyst, such as an inorganic metallic salt catalyst like zinc nitrate, an alkanolamine hydrochloride, like 2-amino-2-methyl-1-propanol hydrochloride and the combination thereof, the function of the latter being to polymerize the ethylene urea, and an emulsifying agent such as an alkylarylpolyether alcohol.

After impregnation, excess emulsion is removed from the textile by any of the aforedescribed means until the resultant wet add-on of emulsion ranges from 50 to 100%, that is, until about from 3 to 15%, by weight, based on dry material, of the condensation product remains on the textile material. The impregnation process may be

repeated two or three times to obtain better penetration and more uniform treatment.

After the excess emulsion has been removed to the proper extent, the impregnated material is dried and cured, either in separate steps or, preferably, in a combined single step, by the application of heat to insolubilize the condensation product. The curing may be carried out, for example, in ovens, with steam cans, tenter frames, hot head presses, and household hand irons. If the drying and curing are performed as separate operations, the drying can be accomplished, for example, in ovens, on steam cans, or on tenter frames, at temperatures from room temperature to 60° C. but preferably below 45° C. Curing, where wrinkle resistance is required, can be accomplished at about from 120° to 220° C. in, for example, ovens, on steam cans, tenter frames, hot head presses, or household hand irons. Where permanent creasing as well as wrinkle resistance is required, as in a garment, the use of hot head presses, hand irons and the like are preferred. It is preferred to dry and cure in a single operation with the hot head presses or hand iron except that it is often desirable with garments containing multiple layers, as in seams, to complete the cure after ironing by hanging the garment in an oven or hot cabinet. The temperature of curing varies, depending upon the curing implement, from 120° to 220° C. The time of curing varies with temperature from ½ minute to 20 minutes, the lower temperature requiring the longer time.

Desired creases dried and cured into the textile material in the aforedescribed manner are permanent to at least 20 launderings. Properly treated fabrics can be subjected to normal laundering and upon dewatering, either with or without centrifugation, will dry free of wrinkles and with retention of the desired permanent creases. Subsequent ironing or pressing is not required.

The following examples are illustrative of the invention. Parts and percentages specified are by weight unless otherwise designated.

EXAMPLE 1

An emulsion was prepared as follows: 29.3 g. of 30% polyethylene emulsion, 0.5 g. of an alkylarylpolyether alcohol as the emulsifying agent, 70 g. of dimethylol cyclic ethylene urea, 7.5 g. of zinc nitrate catalyst, and 405 g. water. Samples of 80 x 80 cotton print cloth (4 oz./sq. yd.) were impregnated with this emulsion and padded to about 75% wet pickup. The samples were then dried and cured in one operation from the wet state by ironing with an electric hand iron set at about 200° C. In curing, creases were set into the samples with the iron and cured in the creased position The samples were then subjected to a repeated laundering test. The laundering consisted of a 20 minutes' sudsing at 140° F. with a detergent in a running suds, followed by rinsing with cold water, centrifugation, and ironing in a flat position to dry. In spite of having been ironed flat the samples returned to their creased state with excellent retention of their original appearance after as many as 12 laundry cycles. Fabrics prepared by this process also have excellent wash and wear properties, drying wrinkle free and retaining their permanent creases after more than 20 standard home-type laundry cycles, without ironing. This example demonstrates that the improved treatment produces a fabric with breaking and tearing strength, wrinkle recovery and crease retention approximately equal to or better than the commercial treatment but with greatly improved resistance to soiling as the higher reflectance readings show.

Table I shows comparative properties of the fabric prepared by the present invention and a fabric prepared by a standard commercial formulation:

Table I

Treatment of Fabric	Breaking Strength (lbs.)		Tearing Strength (lbs.)		Wrinkle Recovery Angle (degree)		Crease Retention	Reflectance Reading[2]
	Warp	Fill	Warp	Fill	Warp	Fill		
Commercial Formulation [1]	29.3	22.6	1.3	0.8	150	152	Excellent	46.4
Formulation of Example 1	28.4	22.8	1.5	0.9	148	145	Excellent	63.1

[1] Contains dimethylol cyclic ethylene urea, acrylic thermoplastic polymer (polyethylacrylate), silicone lubricant (organopolysiloxane), alkylarylpolyether alcohol emulsifying agent and zinc nitrate catalyst.

[2] Reflectance readings were made on samples given an accelerated soiling test consisting of a 7 minute wash at 180° F., in a Launder-Ometer, with 10 mg. carbon black, 0.5 g. yellow soap, and 0.1 g. sodium carbonate per 250 ml. water. The reflectance on untreated fabric was 76.3%.

EXAMPLE 2

Formulation as in Example 1 was used with addition of 10 g. of organopolysiloxane emulsion and 2 g. of metal organic salt catalyst. Physical properties of this sample are essentially equal to that prepared in Example 1 except for soiling propensity. Breaking strength 28.9 lb. (in warp direction), 21.8 lb. (in fill direction); tearing strength 1.5 lb. (warp), 0.9 lb. (fill); wrinkle recovery angle 147° (warp) and 146° (fill); reflectance reading 60.7%; crease retention is excellent.

The process of Example 2 is to demonstrate that a silicone agent can be used with the improved formulation treatment, if desirable, with soiling resistance only slightly less than that shown in Example 1, but with soiling resistance considerably improved over a conventional treatment.

EXAMPLE 3

Cotton trousers (light denim) were impregnated with the formulation in Example 1. Excess emulsion was removed by centrifugation. Final wet add-on was approximately 80%. Drying and curing were performed in one operation by ironing with an electric hand iron set at about 200° C. In curing, creases and pleats were set into the trousers where desired. Creases and wash and wear quality were permanent to more than 20 laundering cycles. This same process is equally applicable to cotton, sport, and dress shirts, jackets, coats, blouses, dresses and the like.

EXAMPLE 4

Garments were given the same treatment as in Example 3 except the formulation of Example 2 was used in place of the formulation of Example 1. The results were the same as those in Example 3. Examples 3 and 4 demonstrate that the processes described in Examples 1 and 2 may be applied to garments.

EXAMPLE 5

80 x 80 cotton print cloth was impregnated with a 12% solution of methylolmelamine containing 5 parts by weight per hundred parts by weight resin of a 35% aqueous solution of 2-amino-2-methyl-1-propanol hydrochloride (an alkanolamine hydrochloride catalyst) and 15 parts by weight of emulsified polyethylene (30% emulsion) per hundred parts by weight of resin. Excess solution was removed by padding and squeezing. Wet pickup was approximately 80%. Drying was performed in a mechanical convection type oven, at 80–90° C. for 5 minutes. Curing was performed in the same oven at 150° C. for 3 minutes. The sample was given a process wash followed by drying. The following table gives the physical properties of this sample compared to a sample treated under identical conditions but without addition of the polyethylene emulsion.

Table II

Treatment of Fabric	Tearing Strength (lbs.)	Abrasion Resistance (cycles)	Wrinkle Recovery Angle (degree)
Methylolmelamine	1.5	150	125
Formulation of this example	1.7	283	134

Example 5 demonstrates that the improved formulation which is the subject of this invention enhances the textile properties of the treated fabric as compared to a fabric treated with the resin only (methylolmelamine resin).

EXAMPLE 6

80 x 80 print cloth was impregnated with a 12% solution of methylated methylolmelamine containing 5 parts by weight per hundred parts by weight resin of a 35% aqueous solution of 2-amino-2-methyl-1-propanol hydrochloride (an alkanolamine hydrochloride catalyst) and from 5–20 parts by weight per hundred parts by weight resin of 30% polyethylene emulsion. Excess solution was removed by padding and squeezing. Wet pickup was approximately 80%. Drying and curing were performed as described in Example 5. Physical properties of samples prepared under this example are tabulated in Table III and compared to a fabric treated under identical conditions but without addition of the polyethylene emulsion.

Table III

Treatment of Fabric	Parts 30% Polyethylene Emulsion per Hundred Parts Resin	Tearing Strength (lbs.)	Abrasion Resistance (cycles)	Wrinkle Recovery Angle (degree)
Methylated Methylolmelamine		1.2	114	129
Formulation of this example	5	1.5	211	138
Do	15	1.5	194	147
Do	20	1.5	279	145

Example 6 shows that the improved formulation which is the subject of this invention enhances the textile properties of the fabric treated as compared to a fabric treated with the same resin only (methylated methylolmelamine).

EXAMPLE 7

This example demonstrates the superiority of the new formulation with respect to resistance to repeated soiling under drastic conditions. Pertinent data is shown in the accompanying table.

Table IV

K/S VALUE ON REPEATED SOILING [1]

	1	2	3	4	5
Untreated Cotton	0.032	0.054	0.063	0.071	0.078
Cotton, treated with dimethylol cyclic ethylene urea	0.014	0.026	0.031	0.035	0.040
Cotton, treated with polyethylene	0.042	0.071	0.084	0.097	0.114
Cotton, treated with dimethylol cyclic ethylene urea-polyethylene	0.071	0.104	0.147	0.153	0.192
Cotton, treated with silicone (organopolysiloxane)	0.141	0.266	0.398	0.487	0.605
Cotton, treated with polyethylacrylate	0.294	0.483	0.639	0.770	0.910
Cotton, treated with dimethylol cyclic ethylene urea-acrylate-silicone	0.243	0.470	0.646	0.801	0.967

[1] Soiling in aqueous solution containing 0.5 g. soap, 0.1 g. Na_2CO_3, and 10 mg. finely divided carbon black per 250 ml., at 180° F. for 7 minutes.

In the above experiment, cotton swatches treated with the indicated formulations were laundered together for 5 successive launderings at 180° C. For the launderings a detergent solution (0.5 g. soap and 0.1 g. sodium carbonate per 250 ml. water) containing a soiling agent (10 mg. fiinely divided carbon black per 250 ml.) was employed.

The data in the foregoing table show that after a single laundering, the untreated cotton control, the cotton treated with dimethylol cyclic ethylene urea resin, the cotton treated with polyethylene only, and the sample treated with resin plus polyethylene all washed essentially clean as shown by visual examination and reflectance determinations. Note however, the cottons treated with silicone only, acrylate only, and the formulation with resin, silicone, and acrylate together, were all dirty after a single laundering. Furthermore, with 5 successive launderings in the presence of the soiling agent the cottons treated with silicone only, acrylate only, and the formulation with resin, silicone, and acrylate together became progressively soiled. The untreated cotton control, the cotton treated with dimethylol cyclic ethylene urea resin, the cotton treated with polyethylene only, and the sample treated with resin plus polyethylene resisted soiling as a result of successive launderings in the presence of the soiling agent.

Even after 5 successive launderings in the presence of the soiling agent, the cotton samples treated with dimethylol cyclic ethylene urea, the cotton samples treated with polyethylene only and the cotton samples treated with resin plus polyethylene were cleaner than were the cottons that represent the standard commercial formulation after the first soiling treatment.

EXAMPLE 8

In this example, a set of cotton samples similar to those in Example 7 were laundered together once in the presence of the soiling agent and then laundered together again 4 more times in succession with the detergent solution but without the soiling agent. (See Table V.)

Table V

K/S VALUE ON REPEATED LAUNDERING [1]

	K/S Value on Soiling [2]	1	2	3	4
Untreated Cotton	0.031	0.017	0.015	0.012	0.008
Cotton, treated with dimethylol cyclic ethylene urea	0.018	0.009	0.007	0.006	0.005
Cotton, treated with polyethylene	0.039	0.026	0.022	0.019	0.016
Cotton, treated with dimethylol cyclic ethylene urea-polyethylene	0.079	0.058	0.057	0.054	0.049
Cotton, treated with silicone (orgahopolysiloxane)	0.133	0.109	0.107	0.105	0.102
Cotton, treated with polyethylacrylate	0.272	0.291	0.312	0.321	0.332
Cotton, treated with dimethylol cyclic ethylene urea-acrylate-silicone	0.314	0.330	0.354	0.367	0.370

[1] Laundering in aqueous solution containing 0.5 g. soap, and 0.1 g. Na_2CO_3 per 250 ml. at 180° F. for 7 minutes.
[2] Soiling in aqueous solution containing 0.5 g. soap, 0.1 g. Na_2CO_3, and 10 mg. finely divided carbon black per 250 ml., at 180° F. for 7 minutes.

The untreated cotton sample, the cotton treated with dimethylol cyclic ethylene urea resin, the cotton treated with polyethylene only, and the sample treated with resin plus polyethylene lost, as a result of repeated launderings, most of what little of the soiling agent they had picked up in the soiling cycle. However, the cotton treated with silicone only, acrylate only, and the formulation with resin, silicone and acrylate together not only resisted cleaning in the successive launderings without the soiling agent but actually picked up some of the soil lost from the improved process samples which were laundered with them.

The tables in Examples 7 and 8 contain data embracing the the "Kubelka-Munk" factors. It has been found (Paul Kubelka, Journal Optical Society of America 38, 448–457 (1948) that there is a quantitative relationship between the reflectance of a fabric sample and the amount of material on the fabric acting to lower the reflectance. The Kubelka-Munk factor or K/S value is calculated from

$$K/S = \frac{(1-R)^2}{2R}$$

where:

R is the observed reflectance
K is the coefficient of reflectivity
S is the coefficient of light scattering

The Kubelka-Munk factors listed in the table give a more accurate picture of actual soiling than reflectance values or visual observation.

We claim:

1. A process of treating a cellulosic textile material to produce a finish thereon which will enable the material to retain permanent creases, exhibit wrinkle-resistance, dimensional stability, shape holding qualities, and resistance to soiling, comprising impregnating the cellulosic textile material with a water emulsion containing, based on the solids content, about from 1 to 15 weight percent of dimethylol cyclic ethylene urea, about 0.15 to 5 weight percent of polyethylene, about from 0.1 to 5 weight percent of a latent curing catalyst, and an emulsifying agent, removing excess emulsion from the impregnated material until the resultant wet add-on of emulsion ranges from about 50 to 100%, and then drying and curing the impregnated material by application of heat to insolubilize the ethylene urea.

2. A process of treating a cellulosic textile material to produce a finish thereon which will enable the material to retain permanent creases, exhibit wrinkle-resistance, dimensional stability, shape holding qualities, and resistance to soiling, comprising impregnating the cellulosic textile material with a water emulsion containing, based on the solids content, about from 1 to 15 weight percent of dimethylol cyclic ethylene urea, about 0.15 to 5 weight percent of polyethylene, about from 0.1 to 5 weight percent of a latent curing catalyst, and an emulsifying agent, removing excess emulsion from the impregnated material until the resultant wet add-on of emulsion ranges from about 50 to 100%, drying the impregnated material at a temperature ranging about from room temperature to 60° C. and curing the dried, impregnated material at a temperature of about from 120° to 220° C. for about from ½ minute to 20 minutes to insolubilize the ethylene urea.

3. A process of treating a cellulosic textile material to produce a finish thereon which will enable the material to retain permanent creases, exhibit wrinkle-resistance, dimensional stability, shape holding qualities, and resistance to soiling, comprising impregnating the cellulosic textile material with a water emulsion containing, based on the solids content, about from 1 to 15 weight percent of dimethylol cyclic ethylene urea, about 0.15 to 5 weight percent of polyethylene, about from 0.1 to 5 weight percent of a latent curing catalyst, and an emulsifying agent, removing excess emulsion from the impregnated material until the resultant wet add-on of emulsion ranges from about 50 to 100%, and simultaneously drying and curing the impregnated material at a temperature of about from 120° to 220° C. for about from ½ minute to 20 minutes to insolubilize the ethylene urea.

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