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[54] **PROCESS FOR PREPARING WASH-WEAR AND DURABLE PRESS COTTONS WHICH WILL ABSORB OPTICAL BRIGHTENERS FROM LAUNDRY DETERGENTS UNDER HOME LAUNDRY CONDITIONS**

4 Claims, No Drawings

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ABSTRACT: Process for preparing a cross-linked cellulosic fabric capable of repeatedly absorbing optical brighteners from wash solution, comprising contacting the said fabric with an aqueous solution comprising a cross-linking agent, about 5–15 weight percent of an alkylpolyethylene oxide alcohol or glycol and thereafter curing the fabric.

PROCESS FOR PREPARING WASH-WEAR AND DURABLE PRESS COTTONS WHICH WILL ABSORB OPTICAL BRIGHTENERS FROM LAUNDRY DETERGENTS UNDER HOME LAUNDRY CONDITIONS

A nonexclusive, 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.

An object of this invention is to impart wrinkle resistance to cellulosic textile materials. More particular, this invention relates to providing a method by which cotton fabrics may be finished so that they will be both wrinkle resistant and yet be able to absorb optical brighteners during laundering. This results in a whiter garment after repeated launderings. As such, this method leads to a more washable product. This property of absorbing optical brighteners from detergents may be produced in fabrics by incorporating in the normal pad solution various amounts of alkylpolyethylene oxide alcohols or glycols.

Heretofore, it is well known that normally cross-linked durable press or wash-wear fabrics have a small capacity to renew their whiteness by absorbing the optical brighteners from everyday laundry detergents. While it is common practice to try to overcome this deficiency by adding the optical brightener to the cross-linking solution, this approach has certain deficiencies. First, the basic chemical nature of many optical brighteners limits their application due to the fact that they tend to neutralize the acid catalyst. This may lead to a poorer durable press appearance. The second limitation is that the high temperatures used in curing may lead to discoloration of the brightener. Finally, with time, discoloration or graying in the fabric may occur due to slow removal of the original optical brightener.

We have now found that by incorporating certain amounts of alkylethyleneoxide alcohols in the pad bath it is possible to produce durable press or wash-wear fabrics which will absorb the optical brighteners in laundering using common detergents. A distinct moiety seems to be required to accomplish this property,

$$R\text{-}[OCH_2\text{-}CH_2]_x\text{-}OH$$

where R is an alkyl group, since neither a long chain alcohol nor a polyethyleneglycol by itself will show this property. The alkylethyleneoxide alcohol or glycol is permanently bound to the fabric by means of a chemical bond via the cross-linking agent. Because of this chemical linkage, the effect is permanent. Thus, after 30 launderings, a cotton fabric treated with a cross-linking formulation containing an alkylpolyethylene oxide alcohol was noticeably whiter than a similar fabric treated with the same formulation without this additive reactant. Another means by which the effect of the alkylpolyether alcohol can be demonstrated is by inspection of the fabrics under ultraviolet light. Under these conditions, a cured fabric prepared from sensitized fabric treated in a conventional manner, was gray. On the other hand, the sample prepared from sensitized fabric treated with a solution containing the alkylethyleneoxide alcohol, was white under ultraviolet light. This reflects the ready absorption of optical brighteners to the latter fabric. These results were readily observed on fabric washes as many as 30 times, thus illustrating the permanent nature of this treatment.

Besides the increased affinity for optical brighteners, the alkylpolyethylene oxide alcohols improve the tearing strength of cross-linked fabric.

In addition to the process outlined above, a variation from contemporary textile practice was also performed. In this case, both the alkylpolyether alcohol and an optical brightener are included with the cross-linking agent in the pad bath solution. The fabric from this treatment looks whiter in regular and ultraviolet light than fabric treated with the same pad bath solution without the alkylpolyether alcohol.

Another variation of these approaches is possible with precured fabric. For this purpose, the precured fabric is given an afterwash in a solution containing an optical brightener. Under these conditions, the washed fabrics treated with cross-linking agent and alkylpolyether alcohol or fabric treated with cross-linking formulation, optical brightener and alkylpolyether alcohol appear whiter than the control fabrics treated with the same formulations, which did not contain any alkylpolyether alcohols.

In general, the process of this invention is the incorporation of these alkylpolyether alcohols in normal pad bath solutions to produce wash-wear or durable press cotton. From this point on, the fabrics are finished in a standard manner according to contemporary textile practice. This method can be used with a number of textile formulations, the only requirement being that a certain amount of water be replaced with an equivalent amount of alkylethyleneoxide alcohol or glycol. For precured fabrics, the modification of using an optical brightener in the afterwash or as a component in a solution in a second afterwash represents some variation from current textile practice on precured fabric.

Alkyl groups from about 4 to about 25 carbon atoms in length have been demonstrated to be effective components of the reactive alcohol moiety. The optimum chain length has appeared to be from about 12 to about 18 carbons in length. The number of ethylene oxide units attached to the alkyl group may be varied over a wide range from about three units to about 25 units and, in general, the ethylene oxide units consist of about from 10 to 80 weight percent of the alcohol. In general, with the preferred alkyl chain length from about 12 to 18 carbon atoms, the preferred number of attached ethylene oxide units appears to be from about three to about nine ethylene oxide units. The preferred structure of the reactive additive would then be

$$C_{12\text{-}18}H_{25\text{-}37}(O\text{-}CH_2\text{-}CH_2)_{3\text{-}9}OH.$$

In addition to this type structure the introduction of side groups in the chain also gives effective additives. In particular, this branching can be accomplished using one or more propylepoxy or butylepoxy alcohols in addition to ethylene oxide as the reactive with the initial alcohol or its ethylene oxide adduct.

The alkylethylene oxide alcohol is effective in pad bath concentrations ranging from about 1 percent to about 25 percent. The preferred range of alcohol concentration for overall performance appears to be from 5 percent to about 15 percent.

The following examples are intended to be representative of treating solutions rather than limiting. Numerous variations are possible for anyone practiced in the textile art.

EXAMPLE 1

A solution was prepared using 8 parts dimethylol dihydroxyethyleneurea, 3 parts of zinc nitrate-based catalyst, 5 parts of alkylethylene oxide alcohol [alkyl carbon chain 12–18 units, 60 percent mol. weight of ethylene oxide units (about 5 ethylene oxide units)], and 74 parts water. This mixture was padded onto fabric, dried 10 minutes at 60° C. and cured for 15 minutes at 130° C. After washing in laundry detergent the fabric was whiter in ordinary light than similar fabric treated with the same pad bath mixture in which the long chain alcohol unit was replaced by water. Under ultraviolet light, the sample with the alcohol additive was white while the control sample was gray.

EXAMPLE 2

The same procedure was followed as in example 1 except that the 5 parts alcohol in the pad bath solution was replaced with 5 parts glycol, with the following type structure:

$R(OC_2H_4)_x\text{-}OCH_2CHOH\text{-}CH_2OH$

The results with the finished fabric and its control prepared without the glycol were similar to those observed in example 1 with the reactive alcohol.

EXAMPLE 3

The same procedure was followed as in example 1. The pad bath contained 8 parts dimethylol dihydroxyethyleneurea 3 parts of zinc nitrate-based catalyst, 15 parts of alkylethyleneoxide alcohol [alkyl group 20–22 carbons in length, 60 percent of total mol. weight as ethylene oxide units (about 8–9 ethylene oxide units)], and 64 parts water. After treatment, the finished fabric showed similar ability to adsorb optical brighteners from laundry solutions as the analogous fabric produced in earlier examples.

The control fabric had a wrinkle recovery angle (WRA) of 281° and a warp tearing strength of 473 g. The alcohol treated fabric had a WRA of 276° and a warp tearing strength of 553 g

EXAMPLE 4

The same procedure was followed as in example 1. The pad bath contained 11 parts dimethylol methoxyethylcarbamate, 3 parts $MgCl_2\text{-}6H_2O$, 5 parts alkylethylene oxide alcohol $C_{12\text{-}18}H_{25\text{-}37}[OCH_2\text{-}CH_2]_{6\text{-}7}\text{-}OH$ and 81 parts water The cure time of fabric treated with this mixture was 5 minutes at 160° C. After washing in laundry detergent, this sample was white under ultraviolet light. By comparison, the control, which was treated with the same mixture except that the 5 parts alcohol was replaced with 5 parts of water, was darker under ultraviolet light.

EXAMPLE 5

A solution was prepared using 6 parts dimethylol dihydroxyethyleneurea, 2.4 parts modified zinc nitrate hexahydrate, 5 parts of polyacrylate, 2 parts silicone, 15 parts alkylethylene oxide alcohol, $C_{12}H_{25}\text{-}C_{18}H_{37}(OCH_2CH_2)_{4\text{-}8}OH$, and 45 parts water. White denim fabric was padded with this mixture. The fabric was dried at 60° C. for 15 minutes. The fabric was fabricated into a trouser cuff, pressed and cured for 15 minutes at 130° C. The cuff was then washed 30 times under normal laundry conditions with a similar cuff prepared in the same manner except that alkylethylene oxide alcohol was replaced with H_2O in the pad bath formulation The cuff, prepared using the pad bath solution containing the alkylethylene oxide alcohol, was whiter in normal light than its control. Furthermore, under ultraviolet light, this cuff was white compared to a gray color for the control.

EXAMPLE 6

A similar procedure was used as in example 1 except that 0.5 parts of an optical brightener was included with the pad bath formulation. After one wash, the fabric so treated was whiter than the control sample in which the alkylethylene oxide alcohol was replaced with an equivalent amount of water. However, it should also be noted that the 0.5 parts of optical brightener was still included in the control treatment.

EXAMPLE 7

This was an analogous treatment to example 4 except that 0.5 parts optical brightener was included in the pad bath formulation. After washing the fabric so treated, it was whiter under ultraviolet light than the fabric with a control treatment using optical brightener but no alkylethyleneoxide alcohol in the formulation.

EXAMPLE 8

A white Oxford was padded with a formulation composed of the following components:

11 parts of dimethylol methoxyethylcarbamate

2.7 parts of magnesium chloride hexahydrate,

15 parts of alkylethyleneoxide alcohol

0.25 parts of optical brightener,

and 61 parts of water. The fabric was dried for 10 minutes at 60° C., fabricated into a cuff, pressed and cured for 5 minutes at 160° C.

A control cuff was also prepared using the same pad bath formulation except that the alcohol was replaced with an equivalent amount of H_2O. After 5 washings, the cuff, prepared from the formulation containing the alkylethyleneoxide alcohol, was whiter than the control cuff.

EXAMPLE 9

Samples of fabrics treated in the manner described in examples 1–4 were washed using wetting agent only. They were then given an afterwash using 0.5 percent optical brightener. The samples which had an alkylethyleneoxide alcohol in the padding formulation adsorbed the brightener and appeared whiter in normal light and white under ultraviolet light. By contrast, the control samples treated without the alkylethylene alcohols were darker under normal light and gray under ultraviolet light.

EXAMPLE 10

A sample of fabric treated in the same manner as example 2, except 12.5 parts of alkylether glycol were used. The fabric was laundered once with a control treated in the same manner except that the alkylether glycol was not in the formulation. Both samples were then treated with an orange dye. The fabric treated with the ether glycol dyed a deep orange while the control was a pale orange

EXAMPLE 11

A solution is prepared using 11 parts dimethylol methylcarbamate, 1 part dimethylol alkylethyleneoxidecarbamate $[R(OCH_2CH)_x\text{-}O\text{-}\overset{O}{\overset{\|}{C}}\text{-}N(CH_2OH)_2]$, 4 parts magnesium chloride hexahydrate and 84 parts H_2O. This mixture is padded onto fabric, dried 10 minutes at 60° C. and cured 5 minutes at 160° C. After several washings in laundry detergent, the fabric is whiter in ordinary light than similar fabric treated with the same mixture without the long chain cross-linking agent. Under ultraviolet light, the sample with the long chain cross-linking agent is white while the control sample is gray.

Similar results are obtained using 9 parts of dimethylol dihydroxyethyleneurea instead of dimethylol methylcarbamate in the cross-linking formulation with and without the long chain cross-linking agent.

We claim:

1. A process for preparing a cross-linked cellulosic fabric capable of repeatedly absorbing optical brighteners from wash solutions, comprising

a. mixing, with a conventional aqueous cross-linking bath containing an acidic catalyst and a cross-linking agent having methylol groups, about from 5 to 15 weight percent of an alcoholic member selected from the group consisting of an alkylethylene oxide alcohol and an alkylethylene oxide glycol and in which member the alkyl group contains about from 4 to 25 carbon atoms and the ethylene oxide units consist of about from 10 to 80 weight percent of the member;

b. padding a cellulosic fabric with the mixture of (a);

c. curing the padded fabric from (b) to effect reaction between the methylol groups of the cross-linking agent and the alcohol groups of the alcoholic member and thereby permanently bind said alcoholic member to the cellulose of the fabric; and

d. washing the cured fabric from (c) free of excess reagents.

2. The product prepared by the process of claim 1.

3. A process for preparing a cross-linked cellulosic fabric containing an optical brightener, comprising:

a. mixing, with a conventional aqueous cross-linking bath containing an acidic catalyst and a cross-linking agent having methylol groups, about from 5 to 15 weight per-

cent of an alcoholic member selected from the group consisting of an alkylethyleneoxide alcohol and an alkylethyleneoxide glycol and in which member the alkyl group contains about from 4 to 25 carbon atoms and the ethylene oxide units consist of about from 10 to 80 weight percent of the member;

b. padding a cellulosic fabric with the mixture of (a);

c. curing the padded fabric from (b) to effect reaction between the methylol groups of the cross-linking agent and the alcohol groups of the alcoholic member and thereby permanently bind said alcoholic member to the cellulose of the fabric; and

d. padding the cured fabric from (c) with a solution containing about 0.5 weight percent of an optical brightener.

4. The product produced by the process of claim 3.