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3,039,167

METHOD FOR IMPROVING THE PROPERTIES OF FABRICS CONTAINING CROSS-LINKED REGENERATED CELLULOSE MATERIAL

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The present invention relates to a method for improving the properties of fabrics containing substantial quantities of cross-linked regenerated cellulose material.

Fabrics made from regenerated cellulose fibers have been in widespread use for many years and have obtained a high level of public acceptance. In the past, however, the kinds of cloth which it has been possible to make, economically, from such fibers have been limited; and in particular it has been difficult if not impossible to manufacture economically goods having a "full" appearance or hand; or having a full, even "cover" such as is normally associated with wool fabrics which have undergone a light fulling operation.

It has now been discovered that fabrics made from regenerated cellulose staple fibers can be given a full hand and a full, even cover by subjecting them to wet agitation, provided that the cellulose fibers have been cross-linked while in the form of staple to the extent of decreasing their water imbibition to not more than about 70%.

The present invention, in a principal aspect, therefore, includes a method for improving the properties of woven and knitted fabrics containing a substantial proportion of regenerated cellulose staple fibers which have been cross-linked while in the form of staple to the extent of reducing their water imbibition to not greater than about 70% which comprises subjecting said fabric to wet agitation.

It has also been discovered that when such fabric, after having been subjected to wet agitation is further treated with a cross-linking agent, its crease resistance is markedly improved, beyond that which would be obtained if the wet agitation had been omitted.

In a second aspect, the present invention, therefore, comprises a process for improving the properties of woven and knitted fabrics containing a substantial proportion of regenerated cellulose staple fibers which have been cross-linked in the form of staple to reduce their water imbibition to not greater than about 70% which comprises subjecting said fabric to wet agitation and subsequently treating the fabric with a cross-linking agent.

In accordance with the present invention, the regenerated cellulose fibers present in the fabric which is subjected to wet agitation are fibers which have been cross-linked while in the form of staple, so that their water imbibition¹ is reduced to not more than about 70% and preferably to between about 30 and 50%.

Fabrics made from such fibers do not degrade under wet agitation, but on the contrary not only retain their strength but, surprisingly, act in a manner somewhat similar to wool, to give a light, soft, full fabric, pleasant to touch and of good appearance without, however, the high degree of shrinkage associated with the fulling of wool fibers.

"Cross-linked" is a term commonly in use in the art to refer to cellulosic fibers in which the cellulose chains are joined or linked together in a direction transverse to their axis by some external agent. An outstanding effect

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of cross-linking is to reduce the water imbibition of the cellulose.

Various agents have been proposed for cross-linking including heat hardenable resinous materials such as the aminoplasts, i.e. heat hardenable condensation products of compounds having at least two amino hydrogens, with methyl forming compounds. Typical aminoplasts are urea-formaldehyde, melamine-formaldehyde, dicyandiamide-formaldehyde, guanidine-formaldehyde and combinations of these ingredients. The term is also used in this application to include the methylol-amino-epihalohydrin compounds described in the copending application of George C. Daul, Serial No. 663,345, filed June 4, 1957, now Patent No. 2,960,484.

Other resinous materials which are suitable for use in the present invention are those formed by the reaction of formaldehyde and acrolein as described in United States Patent No. 2,696,477; resins formed by the reaction of acetone and formaldehyde as described, for example, in United States Patents No. 2,504,835 and No. 2,711,971; polyepoxy resins, e.g. polyfunctional compounds having at least two epoxy groups linked through a hydrocarbon, a polyhydric phenol or a polyhydric alcohol group, such as the resins formed from saturated polyglycidyl ethers of polyhydric alcohols as described in United States Patent No. 2,752,269. Particularly useful compounds of the last named class are the condensation products of epichlorohydrin with ethylene glycols. It will be understood that the precise resin used is not a part of the present invention and other heat hardenable resins used in the textile industry may be employed as desired.

In applying the heat hardenable resinous materials referred to above, it is preferred to use the technique described and claimed in the copending application of G. C. Daul et al., Serial No. 635,695, filed January 23, 1957, now Patent No. 2,902,391.

In place of the heat hardenable resinous materials referred to above, the so-called reactant type resins may be used. Chief among these are the partially or wholly methylated methylolureas and their lower polymers, methylolethylene urea, methylol 1,2-propylene urea and the methyl esters of these cyclic ureas. Such materials may be applied as described in the copending application of Daul Serial No. 819,465, filed June 10, 1959.

Still other cross-linking agents include formaldehyde, polyaldehydes and hydroxyaldehydes, which may be applied as described in the copending application of Daul et al., Serial No. 819,465, filed June 10, 1959.

It should be understood that the above listing of cross-linking agents is not to be intended to be exhaustive and other agents may be used without departing from the invention, provided that the water imbibition of the cellulosic fiber is reduced to below about 70% and provided that this is accomplished by a reaction with the cellulose molecule and not merely by forming a water impervious coating on the outside of the fibers.

In carrying out the wet agitation according to the invention, the aqueous medium may be plain water but preferably includes a wetting agent. The nature of the wetting agent is not especially important. Conveniently it can be common soap, i.e. the alkali metal salts of a mixture of long chain fatty acids. Alternatively, any of numerous synthetic materials having surface active properties can be used including the sulfates and sulfonates of long chain alcohols, e.g. sodium dodecyl sulfate and sodium lauryl benzene sulfonate, alkyl aryl sulfonates, e.g. sodium lauryl benzene sulfonate, sulfated and sulfonated amides and their derivatives, e.g.

Igepon A



¹ Water imbibition is a measure of the amount of water which will be taken up by a fiber. Specifically it is defined as the percent by weight of water retained in saturated material after centrifuging a 0.5 gm. sample for 5 minutes at 1000 G, and is expressed as percent of the oven dry weight of the fiber.

sulfated and sulfonated esters, e.g.

Igepon A $[H_3C(CH_2)_7CH=CH(CH_2)_7COOCH_2CH_2SO_3Na]$

sulfated and sulfonated amines, esters of polyglycerol and fatty acids, glycol esters of fatty acids, triethanolamine soaps of fatty acids and quaternary ammonium salts such as the Sapamines, e.g.



The above list is not intended to be exhaustive, but rather to demonstrate that the type of wetting agent used may vary within wide limits.

The proportion of wetting agent employed is obviously dependent on the type of agent and on the other conditions of the treatment. The more active the agent, the higher the temperature and the longer the time of treatment, the less agent need be used. In general the liquid will contain from 0.2 to 4% wetting agent, by weight. The pH of the liquor should not, however, be above about 12 and is normally between about 6 and about 11.

The temperature of the treatment may also vary greatly depending on the time of treatment and the agent used. Generally it will range from 60° C. to 98° C.

The time of treatment obviously depends on the other variables discussed above. In general it will range from 1/2 to 6 hours.

The amount of liquid which will be used, per pound of fabric, will depend on various factors, including the type of process, the composition of the liquor, the nature of the fabric and the effect desired. In a batch process, normally between about 5 and about 20 pounds of liquor, per pound of fabric, will be used.

In carrying out the treatment, a conventional fulling machine of the type ordinarily used for wool may be employed, or small quantities of fabric may be treated in conventional washing machines. In certain instances the wet agitation may be conducted as a part of a dyeing operation.

The invention is applicable to various types of fabrics including both knitted and woven goods. The fabrics must contain a substantial portion, say above about 50% by weight of regenerated cellulose fiber which has been cross-linked while in the form of staple. Preferably they will contain between about 65 and about 100% by weight cross-linked regenerated cellulose staple fiber. It is preferred that the fabric not contain regenerated cellulose fiber which is not cross-linked. Other fibers which may be used in admixture with cross-linked regenerated cellulose staple fibers include wool, cotton, linen and other natural vegetable and animal fibers, and synthetic fibers including polyester, polyamide, acrylic and cellulose ester fibers. These other fibers may be present in the fabric as an integral part of yarns or threads which also contain cross-linked cellulosic fiber, or they may be present in the fabric as separate yarns or threads not containing cross-linked regenerated cellulose fibers.

After the wet agitation has been completed, the fabric may be washed with clear water to remove the additives present in the fulling bath, if additives were used. It may then be dried and subjected to conventional finishing treatments as desired.

As pointed out above, treatment according to the invention results in a fabric having a full even "cover." The precise effect obtained may be controlled by varying the degree of cross-linking or the severity of agitation, or both, to give a range of materials extruding from those exhibiting a hard worsted-like effect to those exhibiting a soft woolen-like effect.

As noted above, special advantages are obtained if, subsequent to the wet agitation treatment, the fabric is given a further cross-linking treatment.

Final cross-linking treatments of the type referred to are conventionally given to impart crease resistance to fabrics. It is found that when the fabric has been sub-

jected to a wet agitation, in accordance with the present invention, much better crease-resistance can be obtained in the subsequent cross-linking treatment.

The specific conditions under which the cross-linking treatment is carried out will, of course, depend on the nature of the cross-linking agent.

The agent itself has the characteristics described earlier in this specification. Preferably the fabric is passed through an aqueous solution or dispersion of the cross-linking agent and squeezed to retain between about 70 and about 100% of the agent on the fabric, dried and cured at say 130° to 160° C.

In certain cases, it may be desirable to impregnate the fabric with the cross-linking agent, dry at a low (say 60 to 80° C.) temperature and then cure at some later time. This is desirable practice in the case of garments having permanent creases where the fabric must be made into the garment before creases, pleats and the like are put in. By using reactant type resins (see above) and suitable catalysts such delayed curing is possible (see Patent No. 2,846,337).

Again it is possible to make the treated fabrics into garments before impregnating with a crease retaining cross-linking agent. Thus, for example, treated fabric made in accordance with the invention may be cut and sewn to form a garment and the garment pressed to cure the resin and impart permanent creases.

Other variations will readily occur to those skilled in the art.

The invention will be further described with reference to the following specific examples which are given for the purposes of illustration only and are not to be taken as in any way restricting the invention beyond the scope of the appended claims.

Example I

Textile grade rayon staple fiber is impregnated while still in the gel state in accordance with the technique described in Daul et al. Serial No. 635,695, with a liquid containing 5% of an acrolein-formaldehyde precondensate (1 mole of acrolein:4 moles of formaldehyde) 5% of an urea formaldehyde precondensate (1 mole urea:1.6 moles formaldehyde), 0.75% magnesium chloride hexahydrate (catalyst) and 0.15% of a laurel ketene dimer with 0.05% polyglycol stearate (finishing agents). The impregnated fiber is then squeezed to reduce the liquid content to 90% of the weight of the cellulose, dried and cured. It has a water imbibition of 44%. The fiber is then made up into yarn, on conventional textile machinery, and a muslin fabric is manufactured therefrom. A piece of this fabric, 180 by 40 inches, in the loom state is put into a home washing machine with 10 gallons of water at 160° C. to which 0.2% laundry soap is added. The fabric is agitated in the washing machine for 2 hours. At the end of this time, it is rinsed, dried and pressed. It has a soft feel, a light nap and resembles a piece of flannel.

Example II

The fabric processed according to Example I is further impregnated with a solution containing (weight percent):

Dimethylol cyclic ethylene urea	12
Polymethyl methacrylate	1.75
MgCl ₂ ·6H ₂ O	1.20
Water	85.05
	100.00

The fabric is then centrifuged to a solids content of 13%, based on the dry unimpregnated fabric. It is pressed in a hot-head press at 160° C. and further cured at 150° C. for 7 minutes. The fabric has improved crease recovery characteristics compared with the same fabric similarly treated, but not subjected to a wet agitation treatment.

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Example III

Rayon staple fiber is impregnated with a solution having the following composition (weight percent):

Dimethylol cyclic ethylene urea	12
Polymethyl methacrylate	1.75
MgCl ₂ ·6H ₂ O	1.20
Water	85.05
	100.00

The fiber is then made into a 20/1 yarn and woven to a challis fabric on a Draper XD loom. A length of this fabric is run through a conventional fulling machine. The fulling liquor is at 75° C. and contains 0.1% "All" (an alkyl aryl sulfonate manufactured by the Monsanto Chemical Co.). The residence time in the fulling machine is 1.5 hours. After treatment the fabric is rinsed, dried and pressed. It has a soft hand and a light pile.

Example IV

A piece of the fabric processed in accordance with the process of Example III is impregnated with a solution comprising (percent by weight):

Melamine-formaldehyde	6
MgCl ₂ ·6H ₂ O	1.5
Water	Balance

The fabric is squeezed to a 89% wet pick up; then dried and cured at 150° C. The fabric has improved crease recovery characteristics compared to a piece of the same fabric similarly treated, but which had not been subjected to wet agitation.

I claim:

1. A method for improving the properties of woven

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and knitted fabrics containing at least 50% regenerated cellulose fiber which has been chemically cross-linked while in the form of staple to reduce its water imbibition to between about 30% and about 70%, which comprises subjecting said fabric to agitation in an aqueous medium at a pH of between about 6 and about 12, at a temperature between about 60° C. and about 98° C., and for a time between about ½ and about 6 hours, and until a full even cover is developed.

2. The method claimed in claim 1 wherein the aqueous medium contains a wetting agent.

3. A method for improving the properties of woven and knitted fabrics containing at least 50% regenerated cellulose fibers which have been chemically cross-linked while in the form of staple to reduce their water imbibition to between about 30% and about 70%, which comprises agitating said fabric in an aqueous medium at a pH between about 6 and about 12 and at a temperature between about 60° C. and about 98° C. until a full even cover is developed; and subsequently treating said fabric with a cross-linking agent to impart crease resistance thereto.

4. The method claimed in claim 3 wherein the aqueous medium contains a wetting agent.

5. The method claimed in claim 3 wherein the agitation is carried out for from about ½ to about 4 hours.

References Cited in the file of this patent

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