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**PROCESS FOR ANTICREASE FINISHING TEXTILE FABRICS CONSISTING OF CELLULOSE FIBERS AND MIXED FABRICS CONTAINING CELLULOSE FIBERS**

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2 Claims

**ABSTRACT OF THE DISCLOSURE**

Impregnation of cellulose fabric at a pH of below 3 with dimethylol uron or a lower dialkyl ether of dimethylol uron, drying the impregnated fabric to a residual moisture content of between 5 and 25%, based on the dry weight of the fabric, storing the thusly dried fabric for between 1 and 24 hours under conditions excluding air and subsequently rinsing and drying the stored fabric to impart an antcrease finish to the fabric.

This invention relates to a process for antcrease finishing textile fabrics consisting of cellulose fibers and mixed fabrics containing cellulose fibers. More particularly, it relates to an antcrease finish for these fabrics with dimethylol uron and a lower dialkyl ether thereof.

In the wash and wear finishing of textile materials consisting of cellulose fibers or mixed fabrics with N-methylol derivatives of nitrogenous compounds two methods have hitherto been used for cross-linking the cellulose for antcrease finishes.

According to the first method, the fabrics are impregnated with solutions of the N-methylol compounds that further contain an acid donator, such as ammonium chloride, subsequently dried and cured at an elevated temperature for a certain period of time, e.g., 3 minutes at 150° C. This process shall be referred to as "curing process" throughout the application.

According to the second method, which is known from British Pat. 905,044, the textiles impregnated with the finishing agents in an acidic liquor are, immediately after their impregnation batched up and allowed to react whilst still wet. Hereby, a so-called "wet cross-linking" (cross-linking under wet conditions) of the methylol compound with the cellulose fiber takes place. Therefore, this cross-linking process under wet conditions is termed "wet cross-linking process."

By using dimethylol uron or its lower alkyl ethers, however, neither according to the curing nor the wet cross-linking process, wash and wear finishes are obtained that meet all the present-day desiderata. Thus, the textiles finished according to the curing process exhibit a great loss in tensile strength and abrasion fastness, which is greatly adverse to the ulterior stability of the clothing made thereof. The finish achieved according to the wet cross-linking process provides no improvement of the dry-crease resistance, which is required for a favorable wash and wear behavior.

We, now, have found that, using dimethylol uron or its lower alkyl ethers, excellent effects are achieved that are far superior to those finishes hitherto obtained therewith, by effecting the impregnation of the textiles with the solutions of the dimethylol uron or its lower alkyl ethers at pH-values ranging below 3, by subsequently drying the textiles so as to reach a residual moisture content

of between 5 and 25% based on the weight of the completely dried fabric, preferably 8 to 15%, storing them for 1 to 24 hours by exclusion of air, and after storage, rinsing and drying. The latter rinsing operation may also be coupled with a neutralization of the residual acid in the finished fabric by means of, e.g., dilute sodium carbonate solution.

The moisture content of the textile material after the incomplete intermediate drying prior to storage, the so-called residual moisture, is of decisive influence on the obtainable effects. Whereas the wet-crease angles are only little dependent on the residual moisture, the dry-crease angles increases according as residual moisture decreases, which is accompanied by a diminution of the tensile strength. High wet-crease angles and adequate dry-crease angles with little losses in tensile strength are obtained when maintaining, according to the present invention, a residual moisture content of between 5 and 25%, preferably 8 to 15%.

In order to adjust the pH-value of the solutions of dimethylol uron utilizable under the present invention, mineral acids may be employed, such as hydrochloric acid, sulfuric acid and phosphoric acid or also organic acids, such as formic acid or trichloroacetic acid.

Drying of the impregnated material so as to reach the desired residual moisture content may be done at room temperature (about 20° C.) but higher temperatures, e.g. 100° C., may also be used.

The most advantageous storage time for the moist textiles depends on the pH-value of the impregnation liquor and the temperature during storage; the storage time may vary from 1 to 24 hours.

The dimethylol uron utilizable under the present invention is, for example, obtained by saponification of the dimethylol uron dialkyl ethers at a pH-value of 3-2 and temperatures of preferably 50-90° C.

Dimethylol uron dialkyl ethers are prepared in the known manner by short-term heating of urea with formaldehyde under certain conditions and by subsequent etherification of the dried reaction mixture with an alcohol, in the presence of an acid.

The pH-conditions (pH<3) of the finishing process under the present invention are vastly in accord with those of the above-mentioned saponification process (pH 3-2). Therefore, on principle, the ethers of the dimethylol uron may also be employed in place of the dimethylol uron itself, because they are saponified in the prepared liquor. Employing, however, dimethylol uron ether instead of dimethylol uron, somewhat poorer effects are always observed, which might be due to an incomplete saponification.

The processes of the present invention are illustrated by the following examples in which it is to be understood that all temperatures are specified in degrees centigrade, and that the invention is not limited to the specific details of these examples:

**EXAMPLE 1**

A bleached and mercerized fabric consisting of a cotton poplin mixed fabric was impregnated on the padding machine with an aqueous solution containing 150 grams/liter dimethylol uron (in the form of a 60% solution), and 17 grams/liter hydrochloric acid (37% by weight), the pick-up of liquor being 75% based on the weight of the air-dried fabric. Subsequently, the impregnated fabric was dried at a temperature of about 100° so as to reach a residual moisture content of 10%, batched free from creases and wrapped in a plastic sheet, stored for 20 hours, at 20°. At the end of this period, the fabric was rinsed with water, treated with a solution of 2 grams/liter sodium carbonate at 50°, then rinsed again with water, and finally dried at 70°.

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Testing of the technological properties of the finished fabric as compared with the nonfinished one yielded the following results:

	Wet-crease angle		Dry-crease angle		Tensile strength, weft kg.
	Warp	Weft	Warp	Weft	
Finished fabric.....	145°	150°	120°	127°	32.0
Non-finished fabric.....	88°	90°	44°	62°	42.3

The wet-crease angle has been ascertained according to the "Tootal-Method" (see SVF-Fachorgan 12, 754 (1957)), the dry-crease angle according to a draft for DIN 53891 (German Industrial Standards Method 53891) and the tensile strength according to DIN 53801.

As can be seen from the above table high wet-crease resistance properties are achieved, the loss in tensile strength being comparatively low.

#### EXAMPLE 2

A bleached and mercerized fabric consisting of a cotton poplin mixed fabric was impregnated on a padding machine with an aqueous solution that contained, 176 grams/liter dimethylol uron dimethyl ether, and the pH-value of which had been adjusted to 1.3 with hydrochloric acid; the squeezing effect was 74% based on the weight of the air dried fabric. Subsequently, the fabric was dried at 100° to give a residual moisture content of 8%, batched free from creases, and wrapped in a plastic sheet, stored for 16 hours at 20°. It was further treated as described in Example 1.

Testing of the technological properties yielded the following results:

	Wet-crease angle		Dry-crease angle		Tensile strength weft kg.
	Warp	Weft	Warp	Weft	
Finished fabric.....	145°	147°	115°	118°	34.2
Non-finished fabric.....	88°	92°	65°	70°	42.3

Although the dimethylol uron dimethyl ether used in this example has been employed in the same molar concentration as the dimethylol uron in Example 1, lower wet- and dry-crease angles have been achieved with this ether.

If the dimethylol uron dimethyl ether is cross-linked according to a curing process (Example 2 of U.S. Pat. No. 3,048,500)—drying on the pin frame, at 70°, and subsequent heating for 3 minutes to 160°—the finished fabric has shown the following results:

	Wet-crease angle		Dry-crease angle	
	Warp	Weft	Warp	Weft
Finished fabric.....	92°	110°	74°	85°
Non-finished fabric.....	88°	92°	65°	70°

The data on the test of the textile material make obvious the superiority of the "humid cross-linkage" (cross-linkage under humid conditions) according to the present invention.

#### EXAMPLE 3

In order to prove the superiority of the humid cross-linking process under this invention over the wet cross-linking process known from British Pat. No. 905,044 in the wash and wear finishing with dimethylol uron, the following comparative test has been conducted:

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A cotton fabric consisting of a cotton poplin mixed fabric was impregnated on the padding machine with a finishing liquor containing 150 grams/liter dimethylol uron in the form of a 60% solution and 40 cc./liter 12 N-sulfuric acid. In the first case, the impregnated fabric was batched up immediately afterwards, and wrapped in a plastic sheet, stored for 3 hours at 20°. These conditions correspond to the wet cross-linking process.

Another specimen was dried to give a residual moisture content of 7% subsequent to impregnation, and only then batched and stored for 3 hours at 20°, wrapped in a plastic sheet. These conditions correspond to those of the humid cross-linking process under the present invention. In both cases the stored fabrics were subsequently rinsed with cold water, neutralized with a liquor containing 3 grams/liter sodium carbonate, rinsed again with water, and air-dried.

Testing of the finished fabrics yielded the following results:

Cross-linkage conditions	Wet-crease angle		Dry-crease angle	
	Warp	Weft	Warp	Weft
Wet cross-linkage (according to the prior art).....	125°	135°	68°	74°
Humid cross-linkage (according to the invention).....	165°	157°	101°	103°
Untreated fabric.....	88°	92°	76°	79°

The above-given data clearly prove the superiority of the humid cross-linking process under the present invention over the known wet cross-linking process in the finishing with dimethylol uron in particular the small influence of the wet cross-linkage on the dry-crease angles.

It will be understood that this invention is susceptible to further modification and accordingly, it is desired to comprehend such modifications within this invention as may fall within the scope of the appended claims.

What is claimed is:

1. A process for imparting an antcrease finish to a cellulose fiber textile which comprises impregnating said textile with a solution of dimethylol uron at a pH of below 3, drying the impregnated textile to a residual moisture content of between 5 and 25% by weight, based on the weight of the completely dried fabric, then wrapping the thusly dried impregnated textile in a plastic sheet, storing the wrapped fabric for between 1 and 24 hours, subsequently neutralizing excess acid in said fabric and rinsing and drying the thusly treated fabric.

2. The process of claim 1 wherein said residual moisture content is between 8 and 15%.

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