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2,820,715

## MECHANICAL FINISHING OF TEXTILE FABRICS

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This invention relates to improvements in the mechanical finishing of textiles, and is more particularly concerned with improvements in the production of wash-fast effects on fabrics consisting wholly or substantially of regenerated cellulose.

It is well known in principle to impregnate fibres, fabrics and other porous materials with solutions of water-soluble synthetic resins or resin-forming ingredients, then to dry these materials and with the aid of further heating to cure or convert the resin to insolubility. In this manner it is possible to impart to resin-impregnated cellulosic fabrics crease-resistant properties which are permanent and not impaired by washing.

In like manner it has been widely sought to produce on textile fabrics various wash-fast effects by submitting the resin-impregnated materials before the resin is cured to mechanical processing, such as friction-glazing by passing the fabric through heated polishing rolls, or embossing to produce various raised patterns and designs by passing the fabric through heated pressure-rolls which carry the appropriate design in relief. In such cases it has generally been sought to obtain the additional advantages of these wash-fast effects without sacrifice or detriment to the ordinary strength properties and texture or "handle" of the fabrics concerned. Examples of such processes are described in United States Patents Nos. 2,103,293, 2,103,587, 2,148,316, 2,488,397, 2,524,915, and 2,622,995. These processes illustrate the many modifications and attention to detail that have been attempted to achieve useful results.

Whereas urea-formaldehyde resin or resin-forming solutions were originally used, the tendency in industry has more recently been to favour melamine-formaldehyde compositions in such processes because of the enhanced wash-fastness which accrues. However, the greatest care and many precautions have to be exercised in the preparation and use of these compositions for successful working and for avoidance of spoilage and consequent waste of the cloth. It is known in the industry to use methylated melamine-formaldehyde compositions to produce outstandingly good wash-fast mechanical finishes on cotton goods. Surprisingly it has now been found that when rayon or other regenerated cellulose fabrics are similarly processed with methylated melamine-formaldehyde compositions these outstandingly good effects are not realised, and the handle of the material is liable to be adversely affected. The successful development of durable wash-fast finishes on cotton fabrics has created important new fashions and demands for goods therefrom, and it is therefore of considerable importance to the rayon or regenerated cellulose industry that a solution be found to the problem of producing satisfactory mechanical finishes, acceptable to the textile trade, on rayon or other regenerated cellulose fabrics.

The object of the present invention is to solve the above problem.

According to the invention a process for fixing mechanical effects and finishes on textile fabrics consisting

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wholly or substantially of regenerated cellulose, comprises impregnating the fabric with an aqueous solution containing a water soluble lower alkyl ether of dimethylol urea having a minimum etherification of 75% of all the methylol groups present, and an acid or potentially acid curing agent therefor, mangling or otherwise expressing surplus solution from the impregnated fabric, at least partially drying the fabric, subjecting the fabric to the desired mechanical finishing treatment, and heating the fabric to insolubilize the impregnant.

By "regenerated cellulose" we intend to include, inter alia, spun viscose, viscose staple-fibre, viscose continuous-filament yarn and Bemberg or cuprammonium-cellulose yarn.

The solution may contain in addition to the ether of dimethylol urea a lower alkyl ether of a methylol melamine in an amount not greater than the amount of the ether of dimethylol urea. Preferably the methylol melamine contains two or more equivalents of formaldehyde.

After impregnation the material has the surplus solution expressed by mangling or other suitable process so that between 5 and 25% of the ether or ethers based on the weight of the fabric is retained thereon before drying. The fabric is then dried with the avoidance of excessive tension and stretch. Mechanical effects which may be fixed are for example of glazing, embossing and schreinerling.

The preferred ethers are the methyl, ethyl and propyl ethers of dimethylol urea and of methylolmelamines containing two or more equivalents of formaldehyde. The methyl ethers are those generally preferred. In the practice of the invention it is not essential that these ethers be chemically pure entities. Technical products have been found to give quite satisfactory results. Nor is it necessary to isolate these ethers, which are crystalline compounds, in crystalline form. Solutions comprising the alkylated products obtained by reaction of the amino-methylol compounds with the appropriate alcohol may equally well be used, preferably after concentration and recovery of excess alcohol for re-use, but in the use of technical products so described care should be exercised to ensure that the highest possible degree of alkylation has been obtained. Partial alkylation only of the methylol groups present will enhance the water-solubility of the methylol compounds to a mis-leading extent, but the products will be generally less satisfactory since the unconverted methylol groups tend generally towards premature resinification, giving rise inevitably to processing difficulties and harshness of handle in the textile applications.

The following observations are given merely for the purpose of explaining in detail more clearly the improvements and advantages that accrue from the process of the invention.

Rayon fabrics always have an inherently softer drape and handle than cotton or linen of similar construction, and it is of the greatest importance for sales appeal, irrespective of the finishes and effects subsequently incorporated or applied, that these qualities be maintained unimpaired in the finished goods. Natural-fibre fabrics such as cotton goods have already a firmer handle and respond less critically to synthetic resin modification. That is to say, the material and methods of the prior art, which have been designed successfully but primarily for the treatment of natural-fibre fabrics such as cotton and linen, have proved unacceptable when applied to regenerated cellulose, in that the synthetic resin ingredients hitherto used are generally too reactive at too early a stage of the impregnation and drying processes involved, and after drying are contained in the material in a condition retaining insufficient plasticity to respond well to

glazing, embossing or other mechanical finishing, and in all such cases the incomplete or imperfect results achieved are rapidly lost in washing. Attempts to limit or restrict this early condensation or breakdown of the resin by curtailing the drying process are not a satisfactory answer to the problem because, although better plasticity and better mechanical definition accrue from such procedures, wherein the mechanical treatment is carried out at an earlier stage while the impregnated fabric is somewhat damp, such modifications invariably lead to an excessive increase in harshness of handle. Therefore it becomes evident that the problem is one of ensuring that the synthetic resin ingredients contained in the fabric appropriately at the time of subjection to mechanical treatment are precisely and specifically matched to the particular properties and behaviour of the much softer regenerated cellulose, as distinct and very markedly different from natural-cellulose materials.

The synthetic resin solutions employed generally for modifying the properties of textile fabrics are relatively dilute solutions, generally of the order of about 5-25% resin concentration only, necessarily because only minor proportions of synthetic resin in the fabric are required. The behaviour of such dilute solutions during impregnation and drying processes is considerably different from the more viscous and highly concentrated resins used as adhesives for the bonding and laminating of textiles and other materials.

Even in the absence of accelerators or curing agents for the resin, dilute solutions of urea-formaldehyde and melamine-formaldehyde resins or resin-forming ingredients undergo progressive condensation, more rapidly so when curing agents are present, but a further complication less generally recognised is that under heat and during heat-accelerated drying they also undergo appreciable hydrolysis with a marked tendency to loss of formaldehyde. Consequently through changes not only in degree of condensation but also in fundamental composition, the resin remaining in the fabric after drying bears little relationship in properties to the original synthetic resin or resin-forming ingredients originally used for impregnation. The tendency towards progressive condensation is generally well-known. Considerable time and effort have been spent in many of the processes of the prior art to devise improved curing agents having a delayed action, to restrain or minimise this tendency to pre-cure. Few, if any, of these efforts have been completely successful.

It is a property of the alkylated aminoplasts which we utilise and adapt to the process of this invention that the alkylated methylol derivatives of urea and melamine must first be hydrolysed or de-etherified by liberation of their alkyl substituent groups and consequent regeneration of the constituent alcohol and methylol compounds to an appreciable extent before condensation of the methylol compound to resin can begin. Consequently even in the presence of reactive curing agents the resin-forming ingredients are stabilised in solution to a remarkable extent throughout the impregnation and drying processes, by adequate alkylation beforehand.

Even so, the stability and behaviour of the various alkylated compounds we describe are not precisely identical in dilute solutions in various processes. Whereas the alkylated methylol melamines leave after drying a resin of suitable composition and plasticity for satisfactory processing and acceptable results in the mechanical finishing of natural-fibre fabrics, it has unexpectedly been found that when similar materials are applied in practice to textile fabrics consisting of regenerated cellulose either too harsh a handle accrues when the mechanical treatment is applied while the cloth is still damp or, if fully dried, the finishes are imperfect and not fast to washing.

Whereas the effects which accrue from mechanical finishing using melamine compounds are acceptable with cotton and other natural-fibre fabrics and the wash-fast-

ness of these effects leaves nothing to be desired, the use of corresponding alkylated urea compounds in such cases is not generally acceptable because the standard of wash-fastness is much lower.

In contradistinction to these findings with natural-fibre materials, we have found that not only good processing but good wash-fastness acceptable to trade requirements accrue in the case of alkylated dimethylol urea with regenerated cellulose fabrics according to the process of the invention. This good resistance to washing appears to be due to the close matching of the resin plasticity and regenerated cellulose compressibility under friction or pressure in the mechanical treatments applied giving rise to more even distribution of resin in the fibre and a more homogeneous material than has been obtainable hitherto.

If desired, minor adjustments in degree of wash-fastness and draping qualities or handle of the fabric can be made within the scope of the invention by partial replacement of the alkylated methylol ureas with alkylated methylol melamines.

The following examples illustrate the process of the invention:

The methylation of the methyl ether of dimethylol urea used in Examples 1, 3, 4, 7, 8, 9, 10 and 11 was 87 percent of that theoretically possible. That is to say, the percentage of the methoxy groups as determined by the Zeisel method was 36.5 percent, whereas that in a pure dimethyl ether is 41.9 percent. The ethers used in Examples 2, 5 and 6 were laboratory preparations and it is difficult to determine accurately the degree of etherification of these ethers but it is at least 80 percent.

#### Example 1

Viscose rayon staple fabric (Utility 1005) was impregnated with a solution containing 8.5 percent of the dimethyl ether of dimethylol urea and 0.17 percent of ammonium thiocyanate. The fabric was squeezed between mangle rollers so that the solution pick-up was 118 percent on weight of fabric and was dried by circulation of warm air at a temperature below 80° C. The dried fabric therefore contained 10% of its weight of the dimethyl ether of dimethylol urea. The moisture content of the dried fabric was about 10%.

The fabric was embossed under pressure against a heated, embossed metal surface so that a pattern in  $\frac{1}{8}$ " relief was obtained, the temperature during this process being about 130° C. The embossed fabric was then heated for 10 minutes at 140° C. to effect a final cure of the resin formed. The handle of the embossed fabric was excellent and on washing for 10 minutes in 0.5% soap at 95° C., the embossed pattern was unaffected.

#### Example 2

A similar fabric was treated with a solution containing 5% of the diethyl ether of dimethylol urea and 0.1% of ammonium thiocyanate. The pick-up in this case was 120% so that the treated fabric finally contained 6% of reactive material. The procedure of drying, embossing and curing was repeated as in Example 1 and a wash-fast embossed finish was obtained.

#### Example 3

A similar fabric was treated as in Example 1, but in this case the quantity of the dimethyl ether of dimethylol urea was increased so that the treated fabric contained 15% thereof, the quantity of ammonium thiocyanate employed being increased proportionately. The procedure of Example 1 was carried out as before, when the embossed fabric was found to have a good handle and the fastness to washing of the embossed finish was excellent.

#### Example 4

A similar fabric was impregnated with 5% of a mixture consisting of four parts of the dimethyl ether of dimethylol urea and one part of technical trimethyl ether of trimethylol melamine, together with 0.1% of am-

monium thiocyanate. The method of application and the subsequent technique of drying, embossing and final curing was the same as in Example 1. The embossed fabric had an excellent handle and no appreciable loss in definition occurred on washing in 0.5% soap solution for 30 minutes at 95° C.

#### Example 5

A similar fabric was impregnated with 10% of the diisopropyl ether of dimethylol urea and 0.2% of ammonium thiocyanate by the method of Example 1 and similarly dried, embossed and cured. The handle was excellent and the wash fastness of the embossed pattern was good.

#### Example 6

A similar fabric was impregnated with 10% of the di-n-propyl ether of dimethylol urea and 0.2% of ammonium thiocyanate. The ether was not soluble in water at this concentration and 20% of methylated spirits was therefore added to effect solution. The fabric was dried, embossed and cured as before and a satisfactory wash-fast embossed finish resulted.

#### Example 7

The procedure of Example 1 was repeated on a similar fabric except that the bath contained 0.255% diammonium hydrogen phosphate instead of 0.17% ammonium thiocyanate as catalyst. A satisfactory wash-fast embossed finish was obtained.

#### Example 8

A sample of American spun rayon challis having 80 ends and 64 picks per inch was impregnated with 10% of the commercial dimethyl ether of dimethylol urea and 0.3% of tartaric acid. The fabric was dried, embossed and cured as before and an excellent wash-fast embossed finish was obtained.

#### Example 9

300 yards of a printed spun rayon fabric was padded through a solution containing 10% of the commercial dimethyl ether of dimethylol urea, 2% of technical trimethyl ether of trimethylol melamine and 0.188% ammonium thiocyanate.

The fabric was mangled so that the pick-up was 80% and dried on a stenter so as to leave about 10% residual moisture. The dried fabric was passed twice through a glazing calender, the bowls of which were heated to 380° F. and finally cured for 3½ minutes at 300° F. A soft, glazed finish fast to washing was obtained.

#### Example 10

A sample of American spun rayon challis having 80 ends and 64 picks per inch was impregnated with 10% of the commercial dimethyl ether of dimethylol urea and 0.5% of aluminium sulphate. The fabric was dried, embossed and cured as before and an excellent wash-fast embossed finish was obtained.

#### Example 11

Three samples of viscose rayon staple fabric were impregnated with 6% of the commercial dimethyl ether of dimethylol urea and 0.12% of ammonium thiocyanate and dried to various moisture contents as follows:

- A. 1. Normal conditioned moisture of fabric (10% approx.)  
A. 2. 25% moisture  
A. 3. 75% moisture

A further three samples of the same fabric were similarly impregnated with 6% of technical methylated trimethylol melamine and 0.06% ammonium thiocyanate and dried to various moisture contents as shown below:

- B. 1. Normal conditioned moisture of fabric (10% approx.)

- B. 2. 25% moisture  
B. 3. 75% moisture

The samples were then embossed as described in Example 1 and finally cured and examined for handle and fastness of the embossed finish to washing.

The results obtained are summarised in the table below:

Sample ref.	Handle	Fastness to Washing
A. 1. ....	Good. ....	Good. ....
A. 2. ....	do. ....	Do. ....
A. 3. ....	do. ....	Do. ....
B. 1. ....	do. ....	Nil. ....
B. 2. ....	Fair. ....	Poor. ....
B. 3. ....	Harsh. ....	Good. ....

The results obtained using the dimethyl ether of dimethylol urea are therefore independent of the moisture content of the fabric, while those obtained using the technical trimethyl ether of trimethylol melamine are not.

#### Example 12

Two samples of Utility 1005 spun rayon fabric were impregnated with

- (a) 5% methylated dimethylol urea of 85% methylation  
(b) 5% methylated dimethylol urea of 60% methylation

0.1% ammonium thiocyanate was also present in each case (a) and (b).

Both samples were dried in an oven at 85° C., embossed as previously described, cured for 10 minutes at 140° C. and finally washed for 10 minutes in 0.5% soap solution at 95° C.

The sample subjected to treatment (a) showed a good retention of the embossed pattern after washing while the sample subjected to treatment (b) was unsatisfactory.

What is claimed is:

1. A process for fixing mechanical effects on a textile fabric consisting substantially of regenerated cellulose, comprising impregnating said fabric with an aqueous solution containing a water soluble C<sub>1</sub>-C<sub>3</sub> alkyl ether of dimethylol urea having a minimum etherification of 75% of all the methylol groups present and a curing agent therefor, expressing surplus solution from said impregnated fabric, at least partially drying said fabric, subjecting said fabric to the desired mechanical finishing treatment and heating said fabric to insolubilise the impregnant.

2. A process according to claim 1 wherein said solution includes a lower alkyl ether of a methylol melamine in an amount not greater than the amount of said ether of dimethylol urea.

3. A process according to claim 2 in which said methylolated melamine contains at least two equivalents of formaldehyde.

4. A process according to claim 1 wherein 5-25% of said ether based on the weight of the fabric is present therein after surplus solution has been expressed from said impregnated fabric.

5. A process according to claim 2 wherein 5-25% of said ethers based on the weight of the fabric is present therein after surplus solution has been expressed from said impregnated fabric.

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