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FINISHING PROCESS AND COMPOSITION COMPRISING ETHYLENETRIAZINE-UREA-FORMALDEHYDE RESIN, MELAMINE-FORMALDEHYDE RESIN AND UREA-FORMALDEHYDE RESIN

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7 Claims. (Cl. 260—29.4)

This invention relates generally to the production of rayon lining material and more particularly to a finishing process and composition for producing nonshrinking rayon lining material having an improved crease recovery.

Wash-and-wear clothing made of fabrics comprising blends of synthetic yarns and wool or cotton does not shrink from laundering and needs little or no ironing. The use to an increasing extent of such clothing requires an adequate supply of nonshrinking accessories of wash-and-wear quality, such as stiffening material, sewing yarn, and especially lining fabrics. Of the cellulosic fabrics suitable for this latter purpose, cotton and rayon fabrics have been made shrink and crease resistant by finishing them with mixtures of various types of resins. However, these treatments also result in a considerable deterioration of the physical properties of the fabrics. In particular, abrasion resistance and folding number are reduced considerably. Thus, the fabric becomes weak and brittle and consequently useless.

It is therefore an object of this invention to provide a finishing process and composition for producing nonshrink and crease resistant fabrics and yarn which eliminate or at least minimize the aforementioned disadvantages.

Another object of this invention is to provide a finishing process and composition for making cellulosic fabrics shrink resistant and having high crease recovery without substantially reducing the abrasion resistance and folding number of the fabric.

Still another object of this invention is to provide a finishing process and composition for rendering cotton and rayon fabric shrink and crease resistant without causing the fabric to become weak and brittle.

These and other objects will become apparent from the following detailed description.

It has now been found possible to finish rayon and cotton fabrics without appreciably impairing their physical characteristics in a novel, advantageous manner such that their shrinkage does not exceed the permissible limit of about 1% when washed at 40° C. and that are satisfactorily smooth after washing, so that they need not be ironed. In accordance with the invention, this is achieved by applying to the fabric a novel combination of textile finishes and auxiliaries in very critical proportions. The cellulosic fabrics are treated with a composition comprising a mixture of 85–95 grams per liter ethylenetriazine-urea resin, 25–35 grams per liter melamine resin, and a very small amount of an urea resin in an aqueous medium and in the presence of a wash-fast substance which imparts improved hand and firmness, a wetting agent, a wash-fast hydrophobic softening agent, and a diammonium phosphate catalyst. Simultaneously, an organic complexing agent may be used if desired. Surprisingly, an exceptionally good finishing effect not heretofore obtained is produced, resulting in cellulosic fabrics that are very suitable for use as lining material in the manufacture of wash-and-wear clothing.

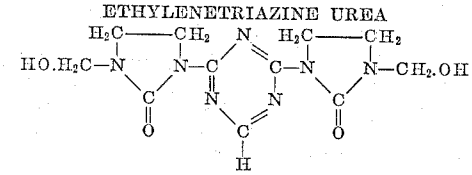
A critical feature of the finish composition is its very low urea resin content which must be maintained between

10 and 12 grams per liter of finishing bath. This is in contrast with known finish compositions using 60 to 80 grams per liter to increase wet strength and shrink-fastness, and 100 to 150 grams per liter with an added catalyst for obtaining crease resistance and a wash-fastness. Surprisingly, the same finishing effects, particularly greater dry crease recovery, are obtained using a fraction of the commonly used amount of urea resin. Moreover, by thus considerably reducing the amount of urea resin, the undesirable action thereof on the fabric is avoided. This is of decisive importance for producing a practical fabric. Furthermore, to obtain a nonshrinking fabric having high wet crease recovery, it is necessary to adhere to the aforementioned critical concentrations of ethylenetriazine-urea resin and melamine resin in the finishing bath. For example, the use of 80 grams per liter ethylenetriazine-urea resin results in fabric having insufficient resistance to shrinkage and poor wet crease recovery.

The desirable effect of the finishing process is attributed to the fact that the various known finishing agents used together surprisingly complement each other and that any unfavorable effects which result from their use are avoided by using very critical amounts. Thus, for example, ethylenetriazine-urea resin by itself produces good resistance to shrinkage and a satisfactory wet crease recovery, but poor dry crease recovery. However, the urea resin necessary to improve the dry crease recovery impairs the other properties of the fabric, particularly, considerably reducing the folding number if used in the customary amount. By reducing that amount, these drawbacks are eliminated and it is still possible when used in combination with ethylenetriazine-urea resin in critical quantities to produce the desired dry crease recovery in the fabric. The melamine component of the mixture further improves the wet crease recovery and favorably influences the hand as well as the wash-fastness of the finishing effect. Unexpectedly, addition of an organic complexing agent to the finish solution contributes to the improvement of the fabric properties, and the presence of a wash-fast substance to impart hand and firmness improves the character of the fabric, particularly the abrasion resistance. Abrasion resistance and hand are also improved by a softening agent, for example, polyethylene imine.

The ethylenetriazine-urea resins used in the present invention, identified by the trade name Knittex CR neu, correspond to the following type formula.

ETHYLENETRIAZINE UREA



The ethylenetriazine ureas are mainly present in the monomolecular phase and react with the cellulose by splitting off water on the methylol groups.

The urea resins utilized in the present invention, identified by the Sandoz carbamide composition Finish EN, belong to the so-called aminoplasts. These compounds are of the synthetic resin type and are obtained by the condensation of urea or thiourea and formaldehyde. Addition of the urea to formaldehyde produces a mixture of monomethylol urea, dimethylol urea, trimethylol urea, and tetramethylol urea. By increasing the amount of formaldehyde added, the composition of the mixture shifts toward the polymethylol compounds. In the desired reaction, one mole of urea is reacted with 2 to 3 moles of formaldehyde. During the formation of methylene bridges, one methylol group reacts with one amino or imino group of the urea while splitting off wa-

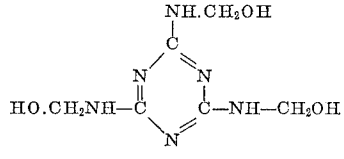
ter. This reaction may result in linear, branched, or cross-linked macromolecules, as is illustrated by the following equations.

$$NH_2.CO.NH.CH_2OH + NH_2.CO- \longrightarrow$$

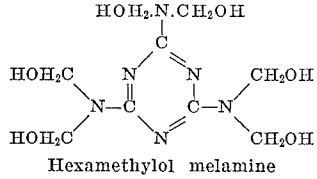
$$NH_2.CO.NH.CH_2.NH.CO- + H_2O - NH.CO.NH.CH_2OH + \underset{\underset{CH_2-}{|}}{NH.CO.NH-} \longrightarrow -NH.CO.NH.CH_2.\underset{\underset{CH_2-}{|}}{N}.CO.NH-$$

A more detailed explanation of the mechanism of the formation of urea resins may be found in Ullmann's Encyklopaedie der technischen Chemie, 1953, vol. 3, page 475.

The melamine resins useful in the present invention are those of the type referred to by the trade name Knittex MM. As in the case of urea, the primary reaction is the addition of formaldehyde to form methylol groups, which continue to react, forming methylene ether bridges in an intermolecular fashion, or methylene bridges with NH_2 or NH radicals. The molar ratios of melamine/formaldehyde are generally between 1:3 to 1:6, with the properties of the resins being more or less dependent upon the number of bonded methylol groups. Although cross-linking and branched polymerization may occur during the formation of the resins, the basic components of the reaction important to the present invention are trimethylol melamine and hexamethylol melamine. The structure of the two compounds is shown by the following formulae:



Trimethylol melamine



Hexamethylol melamine

For further information on the reaction which occurs during the formation of the melamine resins of the present invention, reference may be had to Encyklopaedie der technischen Chemie, 1953, vol. 3, page 487.

The ethylenetriazine-urea resins and the melamine resins used in the present invention, identified by trade names Knittex CR neu and Knittex MM respectively, are products of Chemische Fabrik Pfersee. The urea resin of the present invention, identified by trade name Finish EN, is a product of Sandoz, Inc.

A typical wetting agent which may be used in the present invention is Nekanil LN, an alkylphenol polyglycolether produced by Badische Anilin- and Soda-Fabrik A.G. Typical of washfast hydrophobic softening agents which may be used in the present invention are Primenit VS, an octadecyl ethylene urea produced by Farbwerke Hoechst and Phobol PS (III), a dispersed synthetic resin made by Chemische Fabrik Pfersee and described in Lindner's Tenside-Textilhilfsmittel-Waschrohstoffe, 1964. Particular polyacrylic ester dispersions and polyethylene dispersions which may be used in the present invention are Perapret HV and Perapret TX–1233, respectively, produced by Badische Anilin- and Soda-Fabrik A.G. Depending on the hardness or softness of the water, it may be desirable to use an organic chelating or complexing agent to improve fabric properties. One particular such agent found useful is Trilon B, a sodium ethylenediamine tetra-acetate produced by Badische Anilin- and Soda-Fabrik A.G.

Diammonium phosphate has been found to be a particularly suitable catalyst. Heretofore, magnesium chloride, zinc chloride, or zinc nitrate were customarily used. Such catalysts caused a high decrease in strength of the fabrics in contrast to diammonium phosphate which has only a mild effect, so that the fabric is not damaged as a result of a too violent condensation reaction of the finishing agents. When carrying out the finishing process of the invention, definite conditions must be observed in connection with the pretreatment of the fabric and passage of the fabric through the machine. Particular attention must be paid to the reaction temperature, which must be chosen such that after pre-drying, a certain amount of moisture is left in the fabric.

For purposes of illustrating the invention and not by way of limitation, the following examples are given.

EXAMPLE I

An aqueous finishing bath was prepared containing the following agents per liter of solution.

90 grams ethylenetriazine-urea resin (Knittex CR neu),
30 grams melamine resin (Knittex MM),
10 grams urea resin (Finish EN),
70 grams polyacrylic ester dispersion (Perapret HV),
2 grams wetting agent (Nekanil LN),
0.8 gram sodium ethylenediaminetetra acetate (Trilon B),
8 grams wash-fast hydrophobic softening agent (Phobol PS III),
6 grams diammonium phosphate.

A rayon fabric was conventionally washed, preferably using an anionic detergent and thereafter dyed and dried between 90 and 120° C. Finish was applied to the material by padding on a fowlard with a squeezing effect of about 75%. Subsequently the fabric was dried at about 70° C. such that about 10% of the moisture was retained. Thereafter, the material was cured for eight minutes at 140° C. while in a relaxed state so as to allow the fabric to shrink freely. Subsequently, the cloth was calendered at 40 to 60° C. to obtain a satisfactory hand. Alternatively, a fine material having a good hand may be obtained if after pre-drying and application of the finish, the fabric is calendered and then subjected to curing.

Additional properties of the rayon fabric and the results obtained from the above finishing operation are set forth in the table below as compared with an identical unfinished grey rayon fabric.

Table

Properties and results	Grey rayon fabric	Finished rayon fabric
Yarn-denier/filaments:		
Warp	100/40	100/40
Weft	100/40	100/40
Threads/cm	53/31	53/32
Fabric weight, g./m.^{2}	96	99
Fabric strength, kg./5 cm.(dry)	41. 9/26. 2	48. 8/28. 9
Fabric strength, kg./5 cm. (wet)	20. 9/12. 4	29. 1/16. 3
Relative wet strength, percent	48. 6	58. 5
Elongation, percent (dry)	20. 6	20. 2
Elongation, percent (wet)	28. 3	22. 8
Folding number	3, 890	4, 633
Crease Recovery—Unwashed:		
Dry (DIN 53890) after 5 min	23	51. 5
Dry (DIN 53890) after 60 min	39. 5	71
Wet (Tootal Method) after 3 min	106	140. 5
Dimensional change in percent after mild washing (drum):		
1 wash	−3. 5	+0. 6
2 washes		+0. 6
5 washes		+0. 2
Dimensional change in percent after washing at the boil (drum):		
1 wash		+0. 2
2 washes		−0. 4
5 washes		−1. 4
Degree of swelling, percent	76. 5	52. 1
Change in strength, percent	+0. 5	−12. 3

EXAMPLE II

An aqueous finishing bath was prepared containing the following ingredients per liter of solution.

95 grams ethylenetriazine-urea resin (Knittex CR neu),
35 grams melamine resin (Knittex MM neu),
12 grams urea resin (Finish EN),
50 grams polyacrylic ester dispersion (Perapret HV),
20 grams polyethylene dispersion (Perapret TX 1233),
3 grams wetting agent (Nekanil LN),
8 grams wash-fast hydrophobic softening agent (Phobol PS or Primenit VS),
6.5 grams diammonium phosphate.

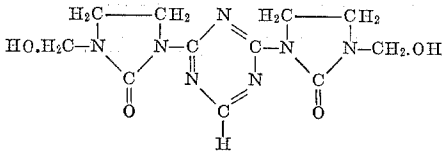
Rayon fabric was treated identically as set forth in Example I, except that the above finish solution was employed. The resultant fabric was shrink resistant and had a high crease recovery without any impairment of physical properties.

From the foregoing examples, it can be seen that applicant's finishing process and composition produces nonshrink fabrics having improved crease recovery without loss in abrasion resistance. The fabrics may be washed in any conventional manner at customary washing temperatures and have an excellent hand after drying. Because of their improved properties, they need not be ironed and thus may be used advantageously as lining material in wash-and-wear clothing.

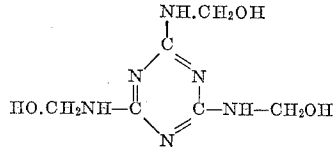
Considerable variations in the process and composition will be apparent to those skilled in the art without departing from the spirit and scope of the invention. It is therefore intended that the invention be limited only as set forth in the following claims.

What is claimed is:

1. A process for producing nonshrinking rayon fabric having a high degree of crease recovery comprising fininshing the fabric with an aqueous solution containing a mixture of 85 to 95 grams per liter of an ethylenetriazine-urea resin having as its principal reactive component molecules of the formula

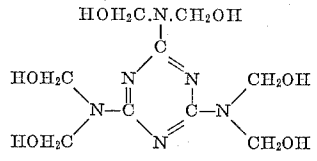


25 to 35 grams per liter of a melmamine resin having as its principal reactive components molecules of the formulae



Trimethylol melamine

and



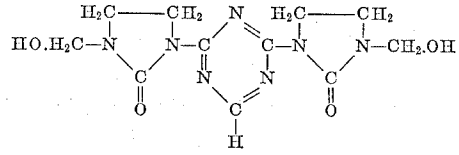
Hexamethylol melamine

and 10–12 grams per liter or a urea resin condensation product of urea and formaldehyde in the presence of a washfast substance to impart hand and firmness, a wetting agent, a wash-fast hydrophobic softening agent and a catalytic amount of diammonium phosphate, drying the fabric to a low moisture content, curing the thus dried fabric, and calendering the fabric to obtain a satisfactory hand.

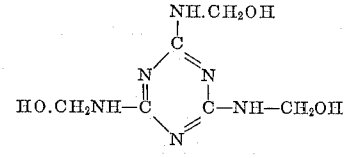
2. The process of claim 1 in which a small amount of an organic complexing agent is present in the finishing solution.

3. The process of claim 2 in which the organic complexing agent is sodium ethylenediamine tetra acetate.

4. A composition of matter comprising an equeous solution containing 85 to 95 grams per liter of an ethylenetriazine-urea resin having as its principal reactive component molecules of the formula

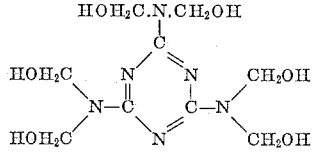


25 to 35 grams per liter of a melmamine resin having as its principal reactive components molecules of the formulae



Trimethylol melamine

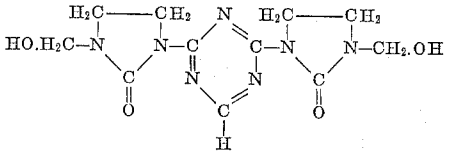
and



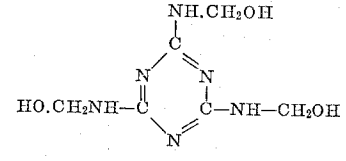
Hexamethylol melamine

and 10 to 12 grams per liter of a urea resin condensation product of urea and formaldehyde.

5. A finishing composition for imparting nonshrinking and improved crease recovery properties to celluosic fabrics comprising an aqueous mixture of 85 to 95 grams per liter of an ethylenetriazine-urea resin having as its principal reactive component molecules of the formula

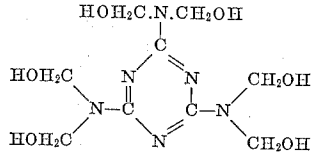


25 to 35 grams per liter of a melmamine resin having as its principal reactive components molecules of the formulae



Trimethylol melamine

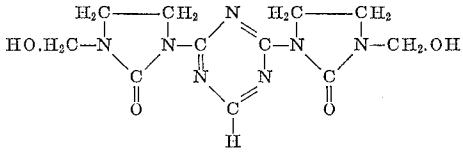
and



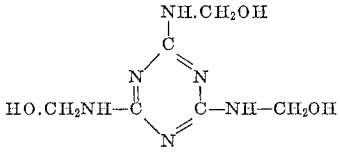
Hexamethylol melamine

and 10 to 12 grams per liter of a urea resin condensation product of urea and formaldehyde.

6. A finishing composition for use in imparting shrink resistance and high crease recovery to cellulosic fabrics comprising an aqueous solution containing 85 to 95 grams per liter of an ethylenetriazine-urea resin having as its principal reactive component molecules of the formula

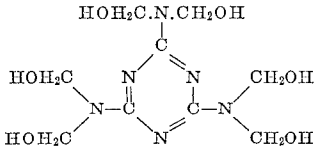


25 to 35 grams per liter of a melamine resin having as its principal reactive components molecules of the formulae



Trimethylol melamine

and

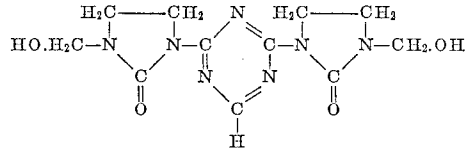


Hexamethylol melamine

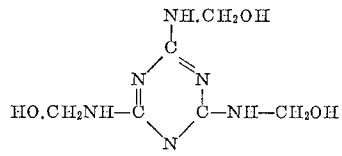
10–12 grams per liter of a urea resin condensation product of urea and formaldehyde, a wash-fast substance for imparting hand and firmness, a wetting agent, a wash-fast hydrophobic softening agent, and a catalytic amount of diammonium phosphate.

7. An aqueous finishing bath for imparting nonshrinking properties and improved crease recovery to rayon fabrics comprising a solution containing 85 to 95 grams per

liter of an ethylenetriazine-urea resin having as its principal reactive component molecules of the formula

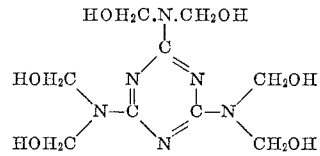


25 to 35 grams per liter of a malamine resin having as its principal reactive components molecules of the formulae



Trimethylol melamine

and



Hexamethylol melamine

10 to 12 grams per liter of a urea resin condensation product of urea and formaldehyde polyacrylic ester dispersion for imparting hand and firmness, a wetting agent, sodium ethylenediamine tetra acetate complexing agent, a wash-fast hydrophobic softening agent and a catalytic amount of diammonium phosphate.

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MURRAY TILLMAN, *Primary Examiner.*