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TREATMENT OF WOOL WITH ACID AMINOTRIAZINE RESIN COLLOIDS

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This invention relates to the treatment of textiles and more particularly to the treatment of wool and wool-containing fabrics particularly to reduce their felting, fulling and shrinking tendencies.

Wool and wool-containing fabrics such as loose wool itself, yarns, threads, woven, felted or knitted cloth have a very undesirable tendency to felt and shrink when subjected to ordinary washing operations. The tendency of these wool materials to felt and shrink is due generally to a curling and intertwining of the wool fibers as the fabrics are wetted and subjected to the mechanical movements of the washing process. As a result the fabric becomes more closely compacted, thicker, and has a considerably reduced area.

A number of different methods have been proposed for the treatment of wool and woolen fabrics to prevent felting and shrinking but all of these have not been completely satisfactory. Some treatments damage the fiber and reduce its wearing qualities, others impart an undesirable hand to the fabric, and some treatments are not permanently effective and may even cause an ultimate increase in shrinkage. Still other shrink-proofing methods are difficult to apply with uniformity and create hazards to the workmen involved in their applications.

One successful method for the treatment of wool to prevent its felting and shrinking is described in the Johnstone and Van Loo U. S. Reissue Patent No. 22,566. According to the process therein described wool-containing fabrics are treated with alkylated methylol melamine solutions and the alkylated methylol melamine is then cured in the fabric to a water-insoluble condition. However, high temperatures of at least about 200° F. and preferably higher are necessary to cure the resin in the fabric.

We have now found that wool and wool-containing fabrics such as unwoven wool, yarns, worsteds, flannels, felts including hat makers felts, blankets, shirting, knit goods and others, either of all wool or of part wool and part silk, viscose rayon, acetate rayon, cotton, etc. may be rendered substantially resistant to felting, fulling and shrinking by the application thereto of a coloidal aqueous solution of a polymerized cationic aminotriazine-aldehyde condensation product containing an acid. It is an advantage of this new process that the high curing temperatures of the Johnstone and Van Loo process referred to above are not required for rendering the resin water-insoluble.

It is an object of the present invention to reduce the felting, fulling and shrinking tendencies of wool and wool-containing fabrics.

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It is another object of the present invention to reduce the felting, fulling and shrinking tendencies of wool and wool-containing fabrics without imparting an undesirable hand to the fabric.

A further object of the present invention is to durably reduce the felting, fulling and shrinking tendencies of wool and wool-containing fabrics.

Still another object of the present invention is to reduce the felting, fulling and shrinking tendencies of wool and wool-containing fabrics without damaging the wool fiber and reducing its wearing qualities.

It is still another object of the present invention to reduce the felting, fulling and shrinking tendencies of wool and wool-containing fabrics without substantially reducing the tensile strength of the wool.

A further object of the present invention is to provide a safe, convenient and effective method for reducing the felting, fulling and shrinking tendencies of wool and wool-containing fabrics.

A still further object of this invention is to improve the wearing characteristics of woolen goods such as durability and resistance to abrasion.

The above and other objects are attained by impregnating wool or a wool-containing fabric with a colloidal aqueous solution of a polymerized cationic aminotriazine-aldehyde condensation product containing an acid and drying the impregnated fabric to convert the aminotriazine-aldehyde condensation product deposited in the fabric to a substantially water-insoluble condition.

The invention will be described in greater detail in conjunction with the following specific examples in which proportions are given in parts by weight unless otherwise indicated. The examples are merely illustrative and it is not intended that the scope of the invention be limited to the details therein set forth.

EXAMPLE 1

100% wool O. D. shirting is treated with a cationic acetic acid colloid of polymerized methylated trimethylol melamine (resin A) by twice immersing the shirting in an 8% resin solids solution and thereafter passing it through suitable rolls, as in a padder, at 3.5 tons pressure so that the wool has a 100% wet pick-up. The shirting is dried to size at 225° F. for six minutes and a portion thereof then dolly washed for five

minutes in a 0.2% solution of N-octadecyl disodium sulfosuccinamate, rinsed for five minutes and then treated for five minutes with a 1% solution of a softening agent which is the reaction product of from 5.5—6.5 mols of ethylene oxide with 1 mol of a mixture of the octadecylamine salt of N-octadecylcarbamic acid and the octadecylguanidine salt of N-octadecylcarbamic acid as described in U. S. Patent No. 2,427,242. After hydroextraction and drying relaxed at 225° F. the tensile strength of the fabric, measured on a Suter tensile tester by the grab method using a 3 inch length, is found to be 60 pounds in the direction of the warp and 50 pounds in the direction of the filling. Untreated shirting, after a five minute light wash and drying, has a tensile strength of 56 pounds in the direction of the warp and 49 pounds in the direction of the filling.

Another portion of the shirting treated as described above with resin A is neutralized at 160° F. with from 5–10% sodium carbonate solution and the so-neutralized shirting and some untreated shirting are given five wool washes as follows: 45 minutes in a reversing wheel washer at 100°–110° F. in a solution of enough neutral soap to give a good running suds, i. e., from about 0.1%–0.25% soap, and three rinses of 5 minutes each with water at 100°–110° F. After the last rinse the shirting is centrifuged, dried loose in a forced circulation oven at 250° F. and then dampened and pressed dry on an electric flat bed press. The warp shrinkage of the treated material is 4.5% while that of the untreated material is 20.5%; the filling shrinkage is 18% for the untreated and 2% for the treated.

EXAMPLE 2

60–40 wool/rayon Fancies are treated in accordance with the general procedure of Example 1 with a 10.5% solids solution of resin A, the pressure on the padder being 4 tons and after the two dips and two nips the material being cured for 9 minutes at 225° F. and dolly washed in 0.1% solution of N-octadecyl disodium sulfosuccinamate.

The warp tensile strength is 35 pounds for untreated material and 31 pounds for the treated, while the filling tensile strength is 22 pounds for untreated material and 18 pounds for the treated. The procedure reduces the warp shrinkage of the material from 46% to 2.5% and the filling shrinkage from 29% to 1.5%.

EXAMPLE 3

100% wool blanket fabric is twice immersed in an 8% solids solution of resin A followed by passing through the rolls of a padder under 2 tons pressure and drying for 9 minutes at 225° F., all in accordance with the general procedure of Example 1. The fabric is given a light dolly wash for 6 minutes; it is then hydroextracted and dried relaxed at 225° F.

The tensile strength of the treated material is 68 pounds in the direction of the warp and 78 pounds in the direction of the filling; corresponding figures for untreated material are 72 and 76. The warp shrinkage for the treated material is 3.5% while that for untreated material is 16.5%. The filling shrinkage drops from 25% for untreated material to 5% for the treated.

EXAMPLE 4

8½ ounce all-wool block plaid is treated generally according to the procedure of Example 1 by twice immersing the wool in an 8% solids solution of resin A and passing through the rolls of a padder under 4 tons pressure. After drying for 6 minutes at 225° F., the fabric is subjected to a light dolly wash for 6 minutes and it is then hydroextracted and dried relaxed at 225° F.

The warp tensile strength for the treated material is 25 pounds whereas that for untreated material is 23 pounds. The filling tensile strength is 19 pounds for the treated material and 20 pounds for untreated. Warp shrinkage of 20% for untreated material drops to 4.5% for treated, and filling shrinkage of 27% for untreated drops to 4.5% for the treated.

EXAMPLE 5

The general procedure of Example 1 is followed and a 100% woolen fabric is twice immersed in an 8.5% solids solution of resin A followed by passage through the rolls of a padder at 4.5 tons pressure. After drying to size for 9 minutes at 225° F. the fabric is dolly washed for 5 minutes in a 0.2% solution of N-octadecyl disodium sulfosuccinamate, extracted for 5 minutes and dried relaxed at 225° F.

The warp tensile strength is 25 pounds for both treated and untreated material and the filling tensile strength is 18 pounds for each. Untreated fabric has a warp shrinkage of 23% and filling shrinkage of 21%; the treated fabric has corresponding shrinkages of 3% and 4.5% respectively.

EXAMPLE 6

The procedure of Example 1 is followed exactly on 100% wool plaid except that after the two dips and two nips the wool is dried to size for 9 minutes instead of 6.

The warp and filling tensile strengths for untreated material are 25 pounds and 19 pounds, respectively. Corresponding figures for the treated material are 24 pounds and 19 pounds. Untreated material has a warp shrinkage of 32% and a filling shrinkage of 26%; the treated material has dropped to 2.5% warp shrinkage and 10% filling shrinkage.

EXAMPLE 7

The procedure of Example 1 is generally followed and a Buffalo plaid of 100% wool, partly reworked, is twice immersed in a 9% solids solution of resin A followed by passing through the rolls of a padder at 1 ton pressure, and then dried to size for 9 minutes at 250° F. The treated plaid is dolly washed for 5 minutes in a solution of N-octadecyl disodium sulfosuccinamate, rinsed for 5 minutes with a 1% solution of a softening agent which is the reaction product of from 5.5–6.5 mols of ethylene oxide with 1 mol of a mixture of the octadecylamine salt of N-octadecylcarbamic acid and the octadecylguanidine salt of N-octadecylcarbamic acid as described in U. S. Patent No. 2,427,242. The material is extracted and dried relaxed at 225° F.

Untreated material has a warp tensile strength of 52 pounds and a filling tensile strength of 31 pounds. Corresponding figures for the treated materials are 44 pounds and 22 pounds, respectively. Warp shrinkage of 25% for untreated material is reduced to 5% for the treated and a filling shrinkage of 23% for untreated material drops to 3% for the treated.

EXAMPLE 8

Again the general procedure of Example 1 is followed and Buffalo plaid woolen shirting fabric

is treated with an 8% solids solution of resin A, squeezed at a pressure of 4.5 tons and dried for 9 minutes at 225° F. The treated fabric is dolly washed for 5 minutes in a 0.1% aqueous solution of the dioctyl ester of sodium sulfosuccinic acid, rinsed for 5 minutes, treated for 5 minutes with the softening agent, hydroextracted and dried relaxed.

The warp tensile strength of untreated material is 41 pounds and of the treated material, 36 pounds. The filling tensile strengths are 47 pounds and 40 pounds, respectively. Warp shrinkage for untreated material is 25% and for the treated, 5%. Filling shrinkage for untreated material is 19% and there is no filling shrinkage for the treated material.

The results of the foregoing examples are set forth in tabular form for convenience:

Ex.	Fabric	Tensile Strength in Lbs.				Shrinkage, Percent			
		Untreated Fabric		Treated Fabric After Neutralization And Light Wash (5 min.)		Untreated Fabric After 5 Wool Washes		Treated Fabric After Neutralization And 5 Wool Washes	
		Warp	Fill	Warp	Fill	Warp	Fill	Warp	Fill
1	100% Wool O. D. Shirting.	56	49	60	50	20.5	18	4.5	2
2	60–40 Wool / Rayon Fancies.	35	22	31	18	46	29	2.5	1.5
3	100% Wool Blanket	72	76	68	78	16.5	25	3.5	5
4	8½ oz. 100% Wool Block Plaid.	23	20	25	19	20	27	4.5	4.5
5	100% Wool	25	18	25	18	23	21	3	4.5
6	100% Wool Plaid	27	19	24	19	32	26	2.5	1
7	100% Wool (partly reworked) Buffalo Plaid.	52	31	44	22	25	23	5	3
8	Buffalo Plaid Woolen Shirting.	41	47	36	40	25	19	5	0

Preparation of resin A

100 parts of 80% aqueous methylated trimethylol melamine prepared in accordance with Example 2 of British Patent No. 566,347 (0.32 mol)
636 parts of water at 80°–90° F.
63.8 parts of glacial acetic acid (1.1 mols)

The methylated trimethylol melamine is completely dissolved by thorough stirring in the water at 80°–90° F. The acid is then slowly added with stirring and the mixture is allowed to age for about 12–16 hours at 70°–90° F. before use. This solution contains 10% resin solids by weight and 76% acetic acid based on resin solids, and may be diluted with cold water as desired. The colloidal solution contains about 3.5 mols of acetic acid per mol of methylated trimethylol melamine.

The colloidal solutions of cationic polymerized aminotriazinealdehyde condensation products containing acids may be applied to the woolen fabrics in various ways known to those skilled in the art. The fabric to be treated is first thoroughly cleaned to remove fats and oils, etc. The dry fabric is then immersed in the resin dispersion and passed through suitable rolls as in a padder or mangle to secure uniform impregnation and to remove excess resin. The fabric, however, may be impregnated by other methods such as, for example, by spraying or with suitable boxes located on the mangle. As our invention is not limited to any particular method of impregnating the wool-containing fabric, other methods will occur to those skilled in the art. Since shrinkage control of woolen fabrics is most effective and economical within the range of about 5 to 15% by weight of resin, based on the dry weight of fabric, the liquor pick-up should be governed accordingly.

After the wool has been impregnated with the alkylated methylol melamine resin colloid, it is dried at temperatures of from about 65°–400° F., or preferably from about 180°–300° F., whereby the resin is deposited in the wool in a substantially water-insoluble condition.

Aminotriazines containing two or three reactive amino groups such as melamine, N-guanylmelamine, ammeline, formoguanamine, acetoguanamine, propioguanamine, phenylguanamine, and N-alkyl-, aryl- and aralkyl-substituted derivatives thereof such as N-methylmelamine, N-phenylmelamine, N-benzylmelamine, etc., which contain a hydrogen atom attached to each nitrogen atom as disclosed in U. S. Patent No. 2,197,357 may be reacted with aldehydes to form the condensation products which are used for preparing the cationic colloidal resin solutions used in the present process. Methylol melamines obtained by condensing melamine with varying amounts of formaldehyde, i. e., in molar ratios of about 1:1 to 1:20 but preferably and generally of about 1:2 to 1:6, and their water-dispersible alkylated derivatives obtained by reacting the methylol melamines with lower aliphatic alcohols such as methanol and ethanol are preferred.

Aldehydes other than the formaldehyde of the examples, such as formaldehyde-yielding materials including paraformaldehyde and hexamethylenetetramine, benzaldehyde, furfural, acetaldehyde, paraldehyde, etc., may be used.

In general the cationic polymerized aminotriazine-aldehyde resin colloidal solutions are prepared in accordance with the teachings of the Wohnsiedler et al. U. S. Patent No. 2,345,543. We prefer using saturated aliphatic monocarboxylic acids having dissociation constants between about 1.4×10^{-5} and 2.5×10^{-4} and water solubilities of at least about 10% for the preparation of the colloidal solutions. Acids included within this category are hydrocarbon and hydroxyhydrocarbon acids as follows:

Acid	Dissociation Constant
Formic	2.1×10^{-4}
Acetic	1.9×10^{-5}
Propionic	1.4×10^{-5}
Butyric	1.5×10^{-5}
Isobutyric	1.5×10^{-5}
Gluconic	2.5×10^{-4}
Glycolic	1.5×10^{-4}
Lactic	1.6×10^{-4}

However, other acids including sulfuric acid, hydrochloric acid, phosphoric acid, and the like, may also be used.

Just as set forth in the Wohnsiedler patent referred to above, the exact amount of acid desirable or necessary for producing the resin colloids is variable. In general we prepare our preferred colloidal polymerized resin solutions using from 0.5–7 mols of the acids set forth above per mol of substantially monomeric aminotriazinealdehyde condensation product. More specifically, in the preparation of preferred colloidal solutions of polymerized methylated methylol melamine resin, we have found it advisable to use about 3 mols of acetic acid or about 1.5 mols of formic acid or from about 2–2.5 mols of glycolic acid for each mol of substantially monomeric methylated methylol melamine.

In order to obtain the desired effect in the present invention wool is so treated with a cationic colloidal aqueous solution of a suitable aminotriazine resin that from about 2.5% to 15%, or preferably from about 5% to about 15%, by weight of resin solids based on the dry weight of the wool will be deposited therein.

We claim:

1. A process which comprises impregnating wool with an acidic colloidal aqueous solution of a polymerized, positively charged, aldehyde condensation product of an aminotriazine having at least two amino groups and a hydrogen atom attached to each amino nitrogen atom, said polymerized condensation product having a definite positive electric charge as shown by its migration toward the cathode on electrophoresis of the solution, said solution containing an amount of acid corresponding to 0.5–7 mols of acid per mol of monomeric condensation product, the take-up of the colloidal solution being such as to deposit in the wool from about 2.5%–15% by weight, based on the dry weight of the wool, of the polymerized aminotriazine-aldehyde condensation product and thereafter drying the impregnated wool whereby the aminotriazine-aldehyde condensation product deposited in said wool is in a substantially water-insoluble condition.

2. A process for reducing the felting and shrinking tendencies of wool-containing fabrics which comprises impregnating a wool-containing fabric with an acidic colloidal aqueous solution of a polymerized, positively charged, aldehyde condensation product of an aminotriazine having at least two amino groups and a hydrogen atom attached to each amino nitrogen atom, said polymerized condensation product having a definite positive electric charge as shown by its migration toward the cathode on electrophoresis of the solution, said solution containing an amount of acid corresponding to 0.5–7 mols of acid per mol of monomeric condensation product, the take-up of the colloidal solution being such as to deposit in the fabric from about 2.5%–15% by weight, based on the dry weight of the fabric, of the polymerized aminotriazine-aldehyde condensation product and thereafter drying the impregnated fabric whereby the aminotriazine-aldehyde condensation product deposited in said fabric is in a substantially water-insoluble condition.

3. A process for reducing the felting-shrinking tendencies of wool-containing fabrics which comprises impregnating a wool-containing fabric with an acidic colloidal solution of a polymerized, positively charged, melamine-formaldehyde condensation product, said polymerized condensation product having a definite positive electric charge as shown by its migration toward the cathode on electrophoresis of the solution, said solution containing an amount of acid corresponding to 0.5–7 mols of acid per mol of monomeric condensation product, the take-up of the colloidal solution being such as to deposit in the fabric from about 2.5%–15% by weight, based on the dry weight of the fabric, of the polymerized melamine-formaldehyde condensation product and thereafter drying the impregnated fabric whereby the melamine-formaldehyde condensation product deposited in said fabric is in a substantially water-insoluble condition.

4. A process as in claim 3 wherein the polymerized melamine-formaldehyde condensation product is polymerized dimethylol melamine.

5. A process as in claim 3 wherein the polymerized melamine-formaldehyde condensation product is polymerized trimethylol melamine.

6. A process as in claim 3 wherein the polymerized melamine-formaldehyde condensation product is polymerized methylated trimethylol melamine.

7. A process for reducing the felting and shrinking tendencies of wool-containing fabrics which comprises impregnating a wool-containing fabric with a colloidal aqueous solution of a polymerized, positively charged, formaldehyde condensation product of melamine, said colloidal aqueous solution containing from 0.5–7 mols of a water-soluble, saturated aliphatic monocarboxylic acid selected from the group consisting of hydrocarbon and hydroxyhydrocarbon acids having dissociation constants between about 1.4×10^{-5} and 2.5×10^{-4} for each mol of substantially monomeric melamine-formaldehyde condensation product and said polymerized condensation product having a definite positive electrical charge as shown by its migration toward the cathode upon electrophoresis of the solution, the take-up of the colloidal solution being such as to deposit in the fabric from about 2.5%–15% by weight, based on the dry weight of the fabric, of the polymerized melamine-formaldehyde condensation product, and thereafter drying the impregnated fabric whereby the melamine-formaldehyde condensation product is deposited in said fabric in a substantially water-insoluble condition.

8. A process for reducing the felting and shrinking tendencies of wool-containing fabrics which comprises impregnating a wool-containing fabric with a colloidal aqueous solution of a polymerized, positively charged, melamine-formaldehyde condensation product containing about 3 mols of acetic acid for each mol of substantially monomeric melamine-formaldehyde condensation product, said polymerized condensation product having a definite positive electrical charge as shown by its migration toward the cathode upon electrophoresis of the solution, the take-up of the colloidal aqueous solution being such as to deposit in the fabric from 2.5%–15% by weight, based on the dry weight of the fabric, of the polymerized melamine-formaldehyde condensation product, and thereafter drying the impregnated fabric whereby the melamine-formaldehyde condensation product is deposited in said fabric in a substantially water-insoluble condition.

9. A process for reducing the felting and shrinking tendencies of wool-containing fabrics which comprises impregnating a wool-containing fabric with a colloidal aqueous solution of a polymerized, positively charged, melamine-

formaldehyde condensation product containing about 1.5 mols of formic acid for each mol of substantially monomeric melamine-formaldehyde condensation product, said polymerized condensation product having a definite positive electrical charge as shown by its migration toward the cathode upon electrophoresis of the solution, the take-up of the colloidal solution being such as to deposit in the fabric from about 2.5%–15% by weight, based on the dry weight of the fabric, of the polymerized melamine-formaldehyde condensation product, and thereafter drying the impregnated fabric whereby the melamine-formaldehyde condensation product is deposited in said fabric in a substantially water-insoluble condition.

10. A process for reducing the felting and shrinking tendencies of wool-containing fabrics which comprises impregnating a wool-containing fabric with a colloidal aqueous solution of a polymerized, positively charged, melamine-formaldehyde condensation product containing about 2–2.5 mols of glycolic acid for each mol of substantially monomeric melamine-formaldehyde condensation product, said polymerized condensation product having a definite positive electrical charge as shown by its migration toward the cathode upon electrophoresis of the solution, the take-up of the colloidal aqueous solution being such as to deposit in the fabric from 2.5%–15% by weight, based on the dry weight of the fabric, of the polymerized melamine-formaldehyde condensation product, and thereafter drying the impregnated fabric whereby the melamine-formaldehyde condensation product is deposited in said fabric in a substantially water-insoluble condition.

11. A process in accordance with claim 3 wherein the impregnated fabric is dried by heating at a temperature of about 225° F.

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