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CELLULOSIC FABRIC FINISHING

Charles D. Stump, Wilmington, Del., assignor to Joseph Bancroft & Sons Company, Rockford, Wilmington, Del., a corporation of Delaware

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This invention relates to textile fabric finishing and is especially concerned with the treatment of cellulosic fabrics, such as cotton, rayon and linen.

It has long been known that various altered characteristics may be imparted to fabrics by processes for treatment with resins or resin-like materials, the effect of many of such treatments being rendered durable, i.e., resistant to laundering and cleaning, by curing or heat treating the resin impregnated fabric. In this way altered characteristics, such as increased crease resistance, increased water repellency and the like may be imparted to various fabrics.

As is also known, various of these treatments, while imparting certain desired characteristics to the fabric also have a tendency to adversely influence certain other characteristics in a manner which is disadvantageous for some uses for which the fabrics are intended. This is especially true with respect to known treatments for imparting good crease resistance to cellulosic fabrics, for instance, cotton fabrics intended for use as shirting material or cotton fabrics intended for use as bed sheets, or nurses' or barbers' uniforms.

In a typical case of a cotton fabric intended for use as a shirting material, the known treatments for crease proofing, for instance, the employment of urea formaldehyde or melamine resins, while imparting substantial crease resistance, at the same time, adversely influence the fabric in certain other respects, notably with respect to what is termed "chlorine retention." In further explanation of this adverse influence, it is noted that the common types of resins employed for crease proofing contain amino groups or nitrogen which rather readily react with chlorine commonly present in bleaching agents used in laundering such fabrics. Such bleaching sometimes directly results in yellowing and loss of strength of the fabric. Moreover, in many cases where immediate yellowing or loss of strength does not occur, these adverse influences occur when the bleached fabric is subsequently heated, for instance, by ironing, the heat of the ironing tending to cause split-off of hydrochloric acid which attacks the cellulose of the fabric and causes undesired yellowing and also weakening of the fabric.

This undesirable "chlorine retention" has long been recognized as a disadvantage of various known crease proofing treatments, and various attempts have been made to diminish chlorine retention. Some improvement in this direction is achieved by the use of dimethylolethyleneurea as the impregnant for the fabric, the improvement apparently being due to the fact that in the molecule of the dimethylolethyleneurea the nitrogen is in part blocked so that linkage with chlorine is reduced.

According to the present invention, a treatment is provided which not only imparts a high degree of crease resistance but completely eliminates chlorine retention. This is accomplished, by the present invention, by the employment in combination of two resins, i.e., a poly-

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these two resins contains nitrogen and therefore neither of them is capable of chlorine retention as described above. However, neither of these resins used alone is capable of achieving the objectives of the present invention for reasons which will be explained herebelow.

First, with respect to the acrylic ester, it has been known that this type of resin will impart some crease resistance to cellulosic fabrics. However, increasing the quantity of the acrylic ester in an effort to attain high crease resistance shows that when the percentage of the ester is increased beyond a moderate value, i.e., beyond about 3.5% by weight of the fabric, the crease resistance progressively diminishes.

With respect to the silicone resins, although it has been known that this type imparts water repellency, this type of resin has not been used for imparting crease resistance. If an attempt be made to increase the quantity of the silicone resin (used alone) in an effort to impart some appreciable degree of crease resistance the water repellency of the fabric is exaggerated to a degree which is undesirable for many fabric uses such for example as for shirting material. In addition, the silicone resin is an expensive ingredient and use of excessive quantities is uneconomical.

I have found, however, that by using the acrylic ester and the silicone resin together, a highly desirable balance of fabric characteristics is achieved including high crease resistance, water absorbency, good hand, high abrasion resistance, and no chlorine retention. In addition to this desirable combination of properties, the fabric treated according to the present invention further shows some increase in crease resistance as a result of at least the initial home launderings. As will further appear, especially advantageous results are secured when employing the two resins in certain quantities or ratios hereinafter set forth.

Beyond the foregoing, I have further found that by the employment of the combination of resins referred to the heating treatment of the impregnated fabric to fix the finish effect may be accomplished in a shorter time and/or at a lower temperature than has been required for various of the prior art crease proofing impregnants. In view of this it is feasible, and the invention contemplates as preferred practice, to use a known type of compressive shrinkage machine for effecting the heat treatment. Thus, with a cotton shirting fabric, the invention contemplates both shrinkage and heat treatment in the same operation, i.e., during the passage of the fabric web through the compressive shrinkage machine. For this purpose either of the well known general types of compressive shrinkage machines may be used, i.e., the Palmer type of machine or the rubber blanket type of machine in each of which the drying drums, rolls or other elements may serve to impart sufficient heat to the impregnated and shrunk fabric to fix the finish effect.

In a typical treatment according to the invention, cotton fabric, for example 136/60 3.60 yards per lb. broadcloth is dipped in a dispersion comprising an aqueous emulsion of a polymerized acrylic ester and a silicone resin. After the dipping, the fabric is squeezed sufficiently to leave a pick-up of from 65% to 75% of the dispersion by weight on the fabric. Thereafter, the fabric is dried to eliminate water, the drying preferably being sufficient to reduce the water to a value below 10 or 15% of the weight of the fabric. This drying may, if desired, be continued sufficiently to render the fabric "bone dry." The fabric web is then passed through a compressive shrinkage machine, for instance, a Palmer type machine with the pressure shoe and feed adjusted to impart the desired degree of shrinkage. The drying drum of the Palmer machine is heated sufficiently to heat the fabric to a temperature between about 200 and 260° F.

When the fabric is delivered from the Palmer machine the finish effect is fixed and durable.

The acrylic esters to be employed according to the present invention are those formed with the lower alkyl alcohols, such as methyl, ethyl and butyl. These resins are available commercially in the form of aqueous emulsion polymers. These resins may also be co-polymerized with minor amounts of certain other materials such as methacrylates, styrene, or acrylonitrile and such co-polymers may also be used provided the secondary constituents are present only in very small quantities, as it is desired to employ a so-called "soft" or textile type of acrylic ester, in order to avoid impairing the hand of the fabric. Acrylic esters of the type here contemplated for use are water insoluble thermoplastics and are also characterized by ability to form somewhat rubbery films.

The silicone resins which are useable according to the invention are polymeric silicone materials having labile hydrogen atoms attached thereto. These silicone resins may be termed methyl hydrogen polysiloxanes and the resin materials which are useable according to the invention comprise aqueous emulsions of such polysiloxanes, constituting oily liquids, which on heating in thin layers yield infusible non-tacky, flexible films. These materials are available in polymerized but relatively low molecular weight form, for instance, a molecular weight of about 1,000 having a viscosity of the order of 1000 to 100,000 centistokes. The silicone resins here contemplated for use are also water insoluble and are thermo-setting to an infusible state under the conditions of heat treating of the impregnated fabric contemplated according to the invention. This general class of silicone resins are also known as textile silicone resins and have heretofore been used in the water proofing of textile fabrics.

Because of the physical characteristics of both of the types of resins described, it is advantageous to use the resins in the form of an aqueous emulsion. The water of the emulsion may or may not contain certain other ingredients in solution therein as will be brought out more fully hereinafter. Although the resins could be applied to the fabric in solution in some organic solvent this technique is not as economically feasible, especially in view of the great simplicity of applying such materials in an aqueous medium.

In preparing the impregnant for application to the fabric, it is convenient to merely intermix aqueous emulsions of the two resin constituents, for instance, the commercially available emulsion form of these resins and then add whatever water is necessary to provide the desired concentration of resins in the dispersion.

The quantity of the acrylic ester present in the impregnating dispersion should be between .5% and 5% by weight and the quantity of the silicone resin should be between .4% and 4% by weight. Especially good results are obtainable where the acrylic ester is used from 1.2% to 3% and where the silicone is used from .5% to 1.25%. Preferably the quantity of the acrylic ester should be from 1.5 to 2.5 times the quantity of the silicone resin by weight. With a pick-up of from 65% to 75% of the dispersion on the fabric, this would provide from .325% to 3.75% of the acrylic ester and from .26% to 3% of the silicone resin on the fabric by weight.

The dispersion preferably also contains some stabilizing agent for the emulsion, for instance, an alkyl aryl polyether alcohol, an example of which is available to the trade under the trademark Triton X-155, marketed by Rohm & Haas Co. A catalyst for the silicone resin constituent may also be present in the dispersion for instance sodium bicarbonate, sodium aluminate, or a metallic salt of a carboxy acid, such as the acetates, hexoates, octoates and naphthenates of lead, iron, zinc, manganese or cobalt, an example of which is available to the market under the trademark Cobelfix marketed by Arkansas Chemical Company. The presence of either the emul-

sion stabilizing agent or the catalyst is not essential to the practice of the invention.

The dispersion may be applied to the fabric either by dipping the fabric or by padding, or by any other convenient technique.

Although some improvement in crease resistance is obtainable merely by impregnating the fabric and then subjecting it to low temperature drying to remove the applied moisture, it is preferred to employ at least some heat treatment after the drying. The temperature of such heat treatment may range from 200° F. to 350° F. from a time ranging from 5 minutes to about 10 seconds. However, since the higher temperatures of this range are not necessary, it is contemplated to utilize a lower range extending from 200° F. to 260° F. A temperature within this latter range may readily be attained during the passage of the fabric through a compressive shrinkage machine, as is contemplated. Even relatively short times within this lower temperature range, for instance, a time from about 10 seconds to about 60 seconds is sufficient to effectively fix the finish and provide good durability.

In the following examples percentages are given on the weight basis unless otherwise indicated.

Example I

A white bleached cotton fabric, 88/80—6.90 yds./lbs., was impregnated with an aqueous dispersion containing the following:

- 1.77% acrylic ester (Rhoplex B-15 emulsion, 46% solids, made by Rohm & Haas Co.)
- 0.72% polysiloxane (Hydro-Pruf emulsion, 30% solids, made by Arkansas Chemical Co.)
- 0.60% catalyst (Cobelfix, made by Arkansas Chemical Co.)
- 0.96% emulsion stabilizer (Triton X-155, made by Rohm & Haas Co.)

The impregnated fabric was squeezed to yield a dispersion pick-up of 65% over and above the dry weight of the fabric, and was then frame dried.

The finished fabric had increased abrasion resistance, high crease resistance, a pleasing hand and did not lose any strength when subjected to a chlorine rinse, water rinse, drying and heating with an iron for 30 seconds at 350° F., according to the Tentative Test Method 69-52 of the 1955 Technical Manual and Year Book of the AATCC. A similar piece of fabric which was treated with 2½% dimethylolethyleneurea lost over 55% of its strength when tested in the same manner. The fabric treated with dimethylolethyleneurea also had considerably lower abrasion resistance.

Example II

A pure bleached cotton broadcloth—136/60, 3.61 yds./lb.—was impregnated and squeezed as in Example I in the dispersion of Example I and dried. After drying, the fabric was run through a rubber blanket compressive shrinkage machine. The crease resistance was increased 30% over the untreated fabric. The abrasion resistance is increased, and there is no loss in the strength of the fabric when it is subjected to the chlorine retention test. A piece of the fabric initially washed in a home type automatic washing machine, had its crease resistance increased an additional 8%.

Example III

A pure bleached cotton poplin fabric, 102/48, was handled as in Example II. After the drying but before passage through the compressive shrinkage machine, the crease resistance of the fabric was moderately increased. After being subjected to the heating in passing through the compressive shrinkage machine the crease resistance was extensively increased—90% greater than the same untreated fabric.

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Example IV

A pure bleached cotton poplin, 102/48, was handled as in Example III, except that the quantity of the silicone resin was reduced to .4% of the dispersion. This resulted in slightly less crease resistance than in Example III, but it still approximated 85% over the untreated fabric.

Example V

A pure bleached cotton print fabric, 80/80, 4.00 yds./lb., was handled as in Example III, except that the quantity of the silicone resin was increased to 2.88% of the dispersion. This also resulted in comparable increase in crease resistance. Tests further show that the concentration of the silicon resin can be further increased to about 4% of the dispersion without appreciable adverse influence on the desired combination of properties of the treated fabric, but such further increase in the concentration ordinarily does not further increase the crease resistance.

Examples VI, VII VIII and IX

This group of examples give comparative results showing the effect of varying the concentration of the acrylic ester. The fabric here treated was a pure bleached cotton print fabric, 80/80, 4.00 yds./lb. which was handled as in Example II. The impregnant here used was the same as in Example I, except for variation in the concentration of the acrylic ester, as follows:

| VI | VII | VIII | IX |
|----------------|-----------------|-----------------|-----------------|
| Percent .88 | Percent 1.77 | Percent 2.65 | Percent 3.54 |

Increase in crease resistance was shown by all of these examples as compared with the untreated in accordance with the following:

| VI | VII | VIII | IX |
|---------------|---------------|---------------|---------------|
| Percent 20 | Percent 42 | Percent 20 | Percent 17 |

Example X

In this example, the same fabric as in Example II was treated in the same way as in that example, except that for the silicone resin (Hydro-Pruf) a similar silicone, Decetex 104, made by Dow-Corning Co. was used. The results were essentially the same.

Example XI

In this example, the same fabric as in Example II was treated in the same way as in that example, except that for the acrylic ester (Rhoplex B-15) a similar acrylic, Rhoplex S-1, made by Rohm & Haas Co., was used. The results again were essentially the same.

Example XII

In this example the fabric and treatment conditions were the same as for Example VII, except that the dispersion contained no emulsion stabilizer and no catalyst. This resulted in a 30% increase in crease resistance, as compared with 42% for Example VII.

Example XIII

This example illustrates the application of the treatment to a linen fabric. An all linen fabric, 42/34, 2.5 yds./lb. was treated with a dispersion according to the

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formulation in Example I. After drying the fabric the crease resistance was increased 21%.

Example XIV

This example illustrates the application of the treatment to a rayon fabric. Here a regenerated cellulose rayon 34/32, 1.82 yds./lb. was treated with the dispersion according to Example I. After drying the fabric, the crease resistance was 29% above the untreated fabric.

From the above examples, it will be noted that the effect of the treatment varies somewhat with different fabrics. In all cases, the increase in crease resistance is good and in no case was there any indication of chlorine retention.

I claim:

1. A method for treating cellulosic fabrics to impart durable crease resistance thereto, which method comprises impregnating the fabric with a dispersion comprising an aqueous emulsion of a polymerized acrylic ester and of a silicone resin, said acrylic ester comprising from .5% to 5% by weight of the dispersion and said silicone resin comprising from .4% to 4% of the dispersion, and thereafter drying the impregnated fabric.

2. A method according to claim 1 in which the quantity of acrylic ester is from 1.2% to 3% and in which the quantity of silicone is from .5% to 1.25%.

3. A method according to claim 1 in which the quantity of acrylic ester in the dispersion is from 1.5 to 2.5 times by weight the quantity of the silicone resin in the dispersion.

4. A method for treating cellulosic fabrics to impart durable crease resistance thereto, which method comprises impregnating the fabric with a dispersion comprising an aqueous emulsion of a polymerized acrylic ester and of a silicone resin, said acrylic ester comprising from .5% to 5% by weight of the dispersion and said silicone resin comprising from .4% to 4% of the dispersion, drying the impregnated fabric to a moisture content below 15%, and thereafter heating the fabric to a temperature of from 200° F. to 350° F. for a time from 5 minutes to 10 seconds.

5. A textile fabric impregnating composition for use in crease-proofing cellulosic fabrics, said composition comprising an aqueous emulsion of from .5% to 5% by weight of a polymerized acrylic ester and from .4% to 4% of a silicone resin, the quantity of acrylic ester being from 1.5 to 2.5 times by weight of the quantity of the silicone resin.

6. A cellulosic textile fabric carrying a crease-proofing agent comprising, by weight of the fabric, a mixture of from .325% to 3.75% of a polymerized acrylic ester and from .26% to 3% of a silicone resin, the quantity of the acrylic ester being from 1.5 to 2.5 times the quantity of the silicone resin by weight.

7. A process for crease-proofing cellulosic textile fabrics by applying an impregnant to the fabric, characterized in that the impregnant applied to the fabric comprises an aqueous emulsion of a mixture of a polymerized acrylic ester and a silicone resin, the quantity of the acrylic ester being from 1.5 to 2.5 times the quantity of the silicone resin by weight.

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