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SHRINKPROOFING TEXTILES WITH VOLATILE POLYAMINES AND EPOXIDES

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A non-exclusive, irrevocable, royalty-free license in the invention herein described, throughout the world for all purposes of the United States Government, with the power to grant sublicenses for such purposes, is hereby granted to the Government of the United States of America.

This invention relates to and has among its objects the provision of novel processes for shrinkproofing and felt-proofing textiles. The invention also includes the provision of improved textiles having superior qualities as to resistance to shrinking and felting. Further objects and advantages of the invention will be evident from the following description.

It is well known in the art that many textile fibers exhibit poor dimensional stability. For example, laundering causes severe shrinkage of woolen textiles. This technical disadvantage seriously restricts the applications of wool in the textile industry and much research has been undertaken in order to modify the natural fiber in order to improve its shrinkage properties. In general, known methods of treating wool to improve its shrinkage characteristics have the disadvantage that the hand of the fabric is impaired, or, if the amount of agent applied is limited to avoid impairment of hand, the degree of shrinkage protection is relatively poor.

In accordance with the invention, wool, or other textile material, is serially subjected to the vapors of a volatile polyamine and a volatile polyepoxide, in any sequence. This procedure yields a product which is virtually shrinkproof under normal laundering procedures while the hand, resiliency, porosity, tensile strength, color, and other valuable properties of the textile are retained. Additionally, the treated textile exhibits a great improvement in resistance to creasing and wrinkling as compared to the original textile.

The prior art discloses various methods for shrinkproofing textiles wherein resins or resin-forming compositions are applied to the fabric. Invariably these procedures involve applying to the textile liquid solutions or dispersions of the resinous materials and usually also applying liquid preparations of curing agents. In the process of the invention the polyamine and polyepoxide in the vapor phase are applied to the textile. Such vapor phase procedure has many significant advantages over conventional liquid phase treatment, as follows:

A primary advantage of the process of the invention is that the vaporous agents can penetrate readily into the textile and ensure uniform formation of an insoluble polyamine-polyepoxide resinous reaction product on all the fibers which make up the fabric. Such uniformity cannot be achieved when the shrinkproofing agents are applied in the liquid phase because of localized variations in the surface properties of the textile, variations in amount of wringing or padding applied to the textile as governed by variations in thickness, density, etc.

Moreover, the vapor phase process of the invention does not disturb the appearance, dimensions, or construction of the textile. There is no change in nap, no wrinkling. Textiles having pleats, folds, etc. retain such forms

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without change. This is in sharp contrast to liquid application procedures wherein such changes are inherent because of the relaxing effects of the liquid on fabric arrangement and construction. Such changes are particularly aggravated by wringing, padding or other applications of mechanical force necessary in liquid phase treatments.

Also, in the process of the invention the original color of the textile is retained. For example, white wool treated in accordance with the invention retains its white color. In the known process as the treated wool tends to develop yellowness. Dyed woolen textiles treated in accordance with the invention retain their original colors in undiminished brightness whereas the processes of the prior art cause a graying or dulling of the fabrics.

The process of the invention yields products of especially soft and resilient properties and more shrinkproofing effect is attained per unit weight of resinous material deposited in the fibers.

In applying the process of the invention, the textile is hung in a chamber where it is exposed to the vapor of either the polyamine or the polyepoxide. The textile is then removed from this chamber and hung in a chamber where it is exposed to the vapors of the other agent (polyepoxide or polyamine). In this treatment, the polyamine and polyepoxide react forming in situ on the fibers a polyamine-polyepoxide resinous reaction product. The vapor phase treatments are continued long enough to deposit on the textile about from 0.5 to 10% of its weight of the resinous reaction product.

To increase the vapor pressure of the polyamine and the polyepoxide—that is, the proportion thereof present within the atmospheres of the respective chambers—the chambers may be heated or evacuated or both heated and evacuated. No special precautions need be taken as to addition or elimination of moisture from either chamber. The temperature within the chambers may be room temperature or may be increased to about 150° C. to get faster treatment. A particular feature of the invention is that a cured resinous deposit—that is, one which is insoluble so that it is not removable from the textile by laundering—is produced even at room temperature. This is desirable since at such lower temperatures, discoloration of the textile is minimized. The time for the treatment will vary depending on such factors as: temperature (higher temperature promotes faster treatment); the nature of the polyamine and polyepoxide selected (more volatile polyamines and polyepoxides under the same conditions will enable faster treatment), and the pressure within the chambers (generally lower pressures will cause more polyamine and polyepoxide to be vaporized, hence the treatment will be faster). As noted above the treatment is complete when the textile has taken up about 0.5 to 10% of its weight of the polyamine-polyepoxide reaction product. Usually, the conditions are so regulated that the major component on the textile is the polyepoxide and of the total polyamine-polyepoxide deposit about 0.5 to 50% is the polyamine. The amount of each agent deposited on the textile can readily be determined by weighing pilot samples of the textiles initially and after subjection to the vapors of each agent. The treatment of the textile may involve, first, treatment with the polyamine then with the polyepoxide, or vice versa. Moreover, the treatment may be repeated to form additional deposit of polyamine-polyepoxide resinous reaction product on the fabric.

The invention is further demonstrated by the following illustrative example:

Pieces of white woolen cloth were placed in a chamber which contained an open vessel of diethylene triamine. The cloth was kept in this chamber for 72 hours, the temperature of the chamber was 25° C. The cloth

was then placed in a chamber which contained an open vessel of butadiene dioxide. This chamber was kept at 105° C. for 3 hours, then reduced to 25° C. and kept at that temperature for 69 hours.

The treated cloth was removed and found to be white in color and to have a soft hand. The pickup of diethylene triamine-butadiene dioxide resinous reaction product was 8.1% based on the weight of the textile.

The treated samples of cloth and a sample of untreated cloth (control) were subjected to tests to determine their shrinkage characteristics. In these tests the cloth samples were subjected to a laundering operation wherein the cloth was violently agitated in an "Accelerator" for 3 min. in a 0.5% solution of sodium oleate at 40° C. with a cloth to solution ratio of 1 to 35. The area of the cloth was measured before and after laundering. The washing tests were carried out in duplicate. The results are tabulated below, the shrinkage values being averages of the duplicate tests.

The results obtained are tabulated below:

Material tested:	Area shrinkage, percent
Treated -----	0
Control -----	54

Butadiene dioxide (also designated 1,2,3,4-diepoxybutane) is preferred as the polyepoxide for use in the process of the invention. However, other volatile polyepoxides may be employed as for example 1,2,5,6-diepoxyhexane, 1,2,4,5-diepoxy-pentane, isoprene dioxide, limonene dioxide. Volatile polyepoxide ethers may also be employed as for example diglycidyl ether, diglycidyl thioether, diglycidyl ether of ethylene glycol, and the like. In general, one may employ any volatile polyepoxide which contains two epoxy



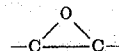
groups and wherein the remainder of the molecule is a carbon chain or a carbon chain interrupted with ether linkages. Preferably the polyepoxides are free from functional groups other than hydroxyl groups, ether groups, and epoxy groups.

Diethylene triamine is preferred as the polyamine for use in the process of the invention. However, other volatile polyamines may be employed as for example ethylene diamine, triethylene tetramine, tetraethylene diamine, 1,4-diamino butane, 1,3-diamino butane, hexamethylene diamine, 3(N-isopropylamino) propylamine, 3,3'-imino-bispropyl amine, etc. In general one can employ any volatile aliphatic polyamine containing at least two amino groups per molecule.

The process of the invention is particularly adapted to the treatment of wool but is also advantageously applicable to other textiles including mohair; animal hair; silk; fibers made from proteins such as zein, casein, peanut protein, soybean protein, keratins, etc.; cotton; regenerated cellulose; viscose; linen; cellulose acetate; etc. The textile material may be in the form of fibers, threads, yarns, woven or knitted fabrics, garments, etc.

Having thus described the invention, what is claimed is:

1. The method of shrinkproofing a textile without significant impairment of its hand which comprises serially exposing the textile to a first agent in the vapor phase and then to a second agent in the vapor phase, to deposit on the textile a total of about from 0.5 to 10% of said agents, based on the weight of the textile, one of said agents being a volatile polyepoxide containing two epoxy



- 30 groups per molecule, the other agent being a volatile aliphatic polyamine containing at least two amino groups per molecule.
2. The method of claim 1 wherein said polyepoxide is butadiene dioxide.
- 35 3. The method of claim 1 wherein said polyamine is diethylene triamine.
4. The method of claim 1 wherein the textile is wool and the polyepoxide is butadiene dioxide.
5. The method of claim 1 wherein the textile is wool and the polyamine is diethylene triamine.

No references cited.