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# TREATMENT OF FABRIC WITH ALKENYLSUCCINIC ACIDS AND ANHYDRIDES TO IMPART WATER REPELLENCY

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9 Claims. (Cl. 117—143)

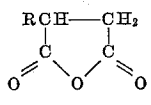
This invention relates to water-repellent cellulosic fabrics and to a method for imparting water-repellency to cellulosic fabrics.

It has long been desirable to have a simple, rapid and economical method of imparting water-repellency to cellulosic fabrics, particularly to various cotton textiles. Many methods of treating fabrics to make them water-repellent have been proposed and have met with varying degrees of success. One method known to the art is to impregnate fabrics with copper salts of certain succinic acid derivatives according to the teachings of U.S. Patent No. 2,381,852. In other methods other hydrophobic materials are placed upon the surface of the fabric. Most of the prior methods have disadvantages in the cost of the treatment or in the time involved in imparting water-repellency to the fabric, and some methods are defective in that certain undesirable characteristics are imparted to the fabric by the various water-repellent agents.

Accordingly, it is an object of the present invention to provide a method of water-proofing cellulosic fabrics. It is a more particular object to provide a method of imparting water-repellency to cellulosic fabrics. It is also an object to provide a cellulosic fabric which is water-repellent. It is a further object to provide a method of imparting water-repellency to fabrics which is both rapid and efficient. It is also an object to provide a method for water-proofing materials which can be carried out at low cost. It is a particular object to provide a cellulosic fabric of desirable characteristics which is also water-repellent.

We have now found that cellulosic textiles, such as cotton, mercerized cotton, linen, etc., can be rendered water-repellent by first impregnating the fabric with an alkenylsuccinic acid or anhydride wherein the alkenyl group contains from 19 to 35 carbon atoms, and then heat-curing the impregnated fabric. The heat-curing step can generally be carried out in less than one hour where temperatures of about 100° C. and over are used.

The alkenylsuccinic acid or alkenylsuccinic anhydride which may be utilized in the present invention includes the alkenyl derivatives of succinic acid or succinic anhydride wherein the alkenyl portion contains from 19 to 35 carbon atoms. The alkenyl substituent in these derivatives is thought to be attached to the succinic acid portion of the molecule at the carbon next to the double bond of the alkenyl group. These derivatives may be represented by the following structural formulas:



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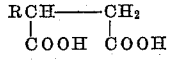


wherein R in each formula is an alkenyl radical having



from 19 to 35 carbon atoms. This group of compounds may be represented by such compounds as 3-eicosenylsuccinic acid and anhydride, 3-heneicosenylsuccinic acid and anhydride, 2-nonadecenylsuccinic acid and anhydride, 11-tricosenylsuccinic acid and anhydride, and 17-pentatriacontenylsuccinic acid and anhydride. The 17-pentatriacontenylsuccinic acid and anhydride and the 11-tricosenylsuccinic acid and anhydride have been found to be the most effective of these derivatives.

The water-proofing compounds described above may be applied to the cloth in any convenient manner. We have found it desirable to disperse or dissolve the agent in water or other solvent and to dip the fabric into the solution or dispersion of the derivative. The function of the solvent therefore is simply to distribute the derivative evenly throughout the fabric; it is immaterial whether the derivative is in the form of a true solution or of a dispersion. The water-insoluble anhydrides may be applied with organic solvents such as isopropanol, benzene, toluene, chloroform, carbon tetrachloride, etc. The dibasic acids of the alkenylsuccinic acids and anhydrides are often dispersible in water but tend to settle out upon standing. The tendency to settle out can be overcome by making the ammonia soap of the dibasic acid and dispersing and/or dissolving such ammonia derivative in water. In some instances the succinic acid and anhydride derivatives can be solubilized not only with ammonia, but also with morpholine or other similar volatile bases, and thus it is possible to avoid the use of organic solvents. We have also found that water emulsions of the treating agents may be used. Thus it is apparent that any media effecting the distribution of the treating agent to the fabric and thus impregnating the fabric with the agent may be utilized.

In water-proofing a cellulosic fabric, the fabric is first freed of contaminants such as wetting and sizing agents or dyes. The fabric is then impregnated with the water-repellent agent by any convenient means such as by dipping the fabric in a solution or dispersion of the chemical. The impregnated fabric is then withdrawn from the solution and air dried. After air drying the cloth is then heated, preferably to a temperature in excess of 100° C., to effect a curing of the impregnated agent within the cloth. We have found that we may conveniently use a temperature of about 125° C. for a period of 15 to 20 minutes. At lower temperatures longer periods of time are required to effect the curing process. To be commercially practical the curing time should be as short as possible and generally less than one hour. At higher temperatures the heat curing may be accomplished in shorter periods of time. The upper limit of temperature at which the heat curing process may be carried out is limited to the temperatures at which fabrics begin to brown or become discolored.

In the method of the present invention, it is preferred to impregnate the fabric with from about 0.7 to 2.5% by weight of the fabric of the alkenyl succinic acid or anhydride. The most practical concentration has been found to be about 2%. Greater amounts of the water-proofing agents may be used, but do not add materially to the water-repellency of the fabric.

The water-repellency of the treated cloth may be evaluated by the spray test method which is described on pages 132–133 of volume 32 of the Technical Manual and Yearbook of the American Association of Textile Chemists and Colorists. This test also is known by A.S.T.M. Designation: D583–54.

In the test procedure, water is sprayed against a taut surface of the fabric sample under controlled conditions to produce a wetted pattern whose size is a measure of the water-repellency of the fabric. The repellency of

the fabric is evaluated by comparing the wetted pattern of the sample with a standard chart. In the standard spray test rating scale, a rating of 100 indicates no sticking or wetting of the upper surface of the sample; 90 indicates a slight random wetting or sticking of the upper surface at spray points; 70 indicates partial or limited wetting of the upper surface; 50 indicates complete wetting of the entire upper surface; 0 indicates complete wetting of entire upper and lower surfaces.

The invention may be further illustrated by the following specific examples:

*Example I*

The following solutions of succinic derivative were prepared:

Solution A—2 gms. dodecenylsuccinic anhydrides in 100 ml. toluene.
Solution B—2 gms. 2-nonadecenylsuccinic anhydride in 100 ml. toluene
Solution C—2 gms. 2(3)-eicosenylsuccinic anhydride in 100 ml. toluene
Solution D—2 gms. 3(4)-heneicosenylsuccinic anhydride in 100 ml. toluene
Solution E—2 gms. 11-tricosenylsuccinic anhydride in 100 ml. toluene
Solution F—2 gms. 17-pentatriacontenylsuccinic anhydride in 100 ml. toluene

Pieces of Indian Head cloth (7½" x 7½") were immersed for 5 minutes in each of the solutions and were then air dried. The impregnated cloths were then heated at 80° C. for 15 hours. The treatment resulted in impregnating the fabric with about 2% (by weight of the fabric) of the various alkenyl succinic anhydrides. The pieces of treated cloth were then tested for water-repellency by the spray test with the following results:

| Cloths tested with: | Standard spray test rating |
|---|---|
| Solution A | 0 |
| Solution B | 70 |
| Solution C | 70+ |
| Solution D | 70+ |
| Solution E | 80 |
| Solution F | 90 |

From this test it is apparent that solutions E and F produced the best results and that water-repellency can be imparted to the fabric at 80° C. where heat-curing conditions are maintained for 15 hours.

The tensile strength of the treated samples of cloth was determined on a Scott upright tester. No substantial difference in tensile strength was found in any sample before or after the treatment.

*Example II*

Six samples were prepared as in Example I except that curing was accomplished by heating each cloth sample for 15 minutes at 115° C. The test results are as follows:

| Cloth treated with: | Spray test rating |
|---|---|
| Solution A | 0 |
| Solution B | 70 |
| Solution C | 70+ |
| Solution D | 70+ |
| Solution E | 80–90 |
| Solution F | 90 |

From this experiment it is apparent that high spray test ratings can be achieved with only a 15 minute heat-curing period if a temperature of 115° C. is employed for the curing process.

*Example III*

Pieces of cotton cloth were immersed in solutions of alkenylsuccinic acids in isopropanol until the fabric became impregnated with alkenylsuccinic acids amounting to 2% by weight of the cloth. The dipped cloth samples were then air dried and cured at 105 to 115° C. for 1 hour. The alkenylsuccinic acids employed in these tests and the spray test ratings obtained therewith are as follows:

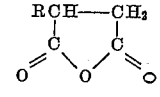
| Acid: | Rating |
|---|---|
| 2-nonadecenylsuccinic acid | 80 to 90 |
| 11-tricosenylsuccinic acid | 80 to 90 |
| 17-pentatricontenylsuccinic acid | 90 |
| 3(4)-heneicosenylsuccinic acid | 80 to 90 |

Equivalent spray test ratings were obtained (in other tests using the same acids with the following solvents) as follows: toluene, ethylacetate, carbontetrachloride, chloroform and mixtures of the straight chain hydrocarbons ordinarily obtained as petroleum fractions or petroleum distillate mixtures having specific boiling ranges.

It is to be understood that the invention described and illustrated above is susceptible of modifications which fall within the spirit and scope of the invention, and that all such variations and modifications are intended to be included within the scope of the appended claims.

I claim:

1. A water-repellent cellulosic fabric impregnated with at least about .75% by weight of a compound selected from the group consisting of



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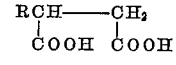

wherein R in each formula is an alkenyl radical having from 19 to 35 carbon atoms.

2. A water-repellent cellulosic fabric as described in claim 1 wherein R is a 17-pentatricontenyl radical.

3. A water-repellent cellulosic fabric as described in claim 1 wherein R is a 11-tricosenyl radical.

4. A cellulosic fabric impregnated with from about .75% to about 2.5% by weight of an alkenyl succinic anhydride wherein the alkenyl group contains from 19 to 35 carbon atoms.

5. A cellulosic fabric impregnated with from about .75% to about 2.5% by weight of an alkenyl succinic acid wherein the alkenyl group contains from 19 to 35 carbon atoms.

6. The method of treating cellulosic fabric to render the same water-repellent comprising impregnating said fabric with a compound selected from the group consisting of an alkenyl succinic acid and an alkenyl succinic anhydride wherein the alkenyl group of each compound contains from 19 to 35 carbon atoms, and heat-curing the impregnated fabric at temperatures lower than the temperatures at which the cellulosic fabric is discolored.

7. The method of treating cellulosic fabric to render the same water-repellent comprising impregnating said fabric with a compound selected from the group consisting of an alkenyl succinic acid and an alkenyl succinic anhydride wherein the alkenyl group of each compound contains from 19 to 35 carbon atoms, and heat-curing the impregnated fabric at temperatures in excess of about 100° C. and lower than the temperature at which the cellulosic fabric is discolored for a period of from about 10 to 60 minutes.

8. The method of making a cellulosic fabric water-repellent comprising impregnating said cellulosic fabric

with from about .75% to about 2.5% of an alkenyl succinic acid having from 19 to 35 carbon atoms in the alkenyl group thereof, and heating the impregnated cellulosic fabric in air at a temperature in excess of about 100° C.

9. The method of making a cellulosic fabric water-repellent comprising impregnating said cellulosic fabric with from about .75% to about 2.5% of an alkenyl succinic anhydride having from 19 to 35 carbon atoms in the alkenyl group thereof, and heating the impregnated cellulosic fabric in air at a temperature in excess of about 100° C.



**References Cited** in the file of this patent

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