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WATER-REPELLING COMPOSITION

Norman G. Mailander, Racine, Wis., assignor to S. C. Johnson & Son, Inc., Racine, Wis.

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This invention relates to the treatment of fabrics to render them water-repellent. More particularly, the invention relates to solutions of certain waxy materials and novel water-repelling agents, substantive to fabrics and capable of imparting water-repellent effects to them.

Materials and methods advocated by prior art to impart water-repellency to fabrics have been sundry and numerous. Whereas a comparatively high degree of resistance has been effected upon wool with compositions of both the aqueous dispersion and non-aqueous solution varieties, such has not been accomplished without a subsequent curing operation with other extensively used fabrics, especially cotton. It has been found that cotton may be transmitted a satisfactory degree of repellency when treated with certain aqueous dispersions or with what is known in the trade as a "laundry type" composition. However, no material or composition has been disclosed which has proved satisfactorily operable upon cotton material when such is dry cleaned. Nevertheless, the latter method of cleaning cotton garments which require water-repellency (raincoats, jackets, etc.) is preferred, since the laundering of such garments causes shrinkage, color fading and subsequent difficult ironing or pressing.

It is therefore the object of this invention to provide a composition which will offer not only an optimum degree of water-repellency to wool, but will permit the dry cleaning of such fabrics as cotton and result in an heretofore unattained repellency of the fabric without the necessity of subsequent curing.

This novel composition is a solution comprising from 1 to 10% by weight of the material selected from the group consisting of petroleum waxes, petrolatums, polymerized octadecyl vinyl ether and mixtures thereof, an alkoxide selected from the group consisting of aluminum and zirconium alkoxides having from 3 to 18 carbon atoms and mixtures thereof, and a mutual solvent for said material and said alkoxide. The solvent constitutes the major portion by weight of said solution.

In applicant's co-pending application, S. N. 183,916, water-repelling compositions are formed with zirconium ethoxide.

Now, having indicated in a general way, the nature and purpose of this invention, the following examples will illustrate the invention. It is understood, however, that such examples are presented merely as illustrations of the invention, and are not to be construed as limiting the

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same. In the examples, the ingredients are given in parts by weight, unless otherwise indicated.

Example 1

Forty parts of refined paraffin wax were dissolved in 764.4 parts of Stoddard solvent by heating the mixture in a suitable vessel to a temperature of approximately 120° F. In a second vessel, 21 parts of aluminum isopropoxide and 3.6 parts of octylene glycol were dissolved in 166 parts of Stoddard solvent by heating to a temperature of approximately 230° F. while the mixture was simultaneously agitated. At this stage, the solution appeared clear and was combined with the ingredients of the first vessel and allowed to cool to approximately room temperature. A swatch of wool gabardine and a swatch of cotton twill material were dipped in the cooled solution and remained immersed therein for a period of two minutes after which they were withdrawn from the solution bath and the liquid constituents of the composition withdrawn from the swatches by means of centrifuging. The swatches were then exposed to the atmosphere for a period of five to six hours and upon drying were steam pressed in a manner simulating commercial pressing methods. The swatches were then submitted to a spray test to determine the degree of water-repellency. The test apparatus consisted of a 6-inch glass laboratory funnel held by a laboratory ring support. A spray nozzle connected to the funnel tube by a piece of ⅜-inch rubber tubing, was provided with 20 holes, drill Number 65, 0.035-inch in diameter. The nozzle was supported directly over the center of an embroidery hoop mounted on a block of wood, so that the plane of the swatch held on the hoop made an angle of 45° with the horizontal. The distance of the nozzle to the center of the hoop-mounted swatch was 6 inches.

250 ml. of water having a temperature of 80°±1° was quickly poured into the funnel and allowed to spray down upon the fabric. The duration of the spraying was approximately 25 to 30 seconds. Upon completion of the spraying period, the hoop was grasped at one edge and the other edge tapped against a solid object, the fabric facing this object during tapping. The hoop was then turned 180°, grasped at the opposite end and tapped at the point previously held. The swatch was then examined and designated a spray rating. Both the cotton twill and the wool gabardine swatch indicated a rating of 100.

A solution was formulated identically with that set forth above except that 21 parts of zirconium

isopropoxide was substituted for the aluminum isopropoxide. Wool swatches dipped in the solution and subjected to the spray test evidenced a spray rating of 100 while cotton swatches indicated a rating of 90.

Example 2

Forty parts of refined paraffin wax were melted by heating to a temperature of approximately 135° F. and in this melted condition were added to 934 parts of naphtha. Twenty-six parts of aluminum isopropoxide were then mixed with the wax solution and dissolved therein. A portion of the composition was then placed in a charge cup of a siphon type pressure spray from which the solution was sprayed upon two swatches, one of wool and the other of cotton, with a gun pressure of approximately 25 lbs. p. s. i. The spray was moved over the swatches at a rate sufficient to allow visual notice of wetting caused by the delivery of the composition upon the fabric. The swatches were then dried and pressed in a manner similar to that used in commercial pressing establishments and thereupon submitted to spray rating tests identical to those disclosed in Example 1 and it was indicated that both the cotton and wool swatches possessed a spray rating of 100.

Twenty-six parts of zirconium isopropoxide replaced the aluminum alkoxide in a solution otherwise identical to that above and both wool and cotton swatches sprayed therewith bore a spray rating of 100.

Example 3

A water-repellent composition was formulated having 20 parts of aluminum isopropoxide and 5 parts of refined paraffin wax, and 975 parts naphtha. Cotton swatches were dipped in the material and subsequently treated as set forth in Example 1. Other cotton swatches were sprayed as disclosed in Example 2. Then the treated swatches were submitted to a spray rating test specified in Example 1 and it was found that as a result of both the spray and dip method, the fabric possessed a spray rating of 50.

A solution was formulated similar to that above except that 20 parts of zirconium isopropoxide was the alkoxide used. Cotton swatches treated with the solution by either dipping or spraying indicated a resulting spray rating of 50.

Example 4

A solution was prepared with 30 parts of aluminum octadecoxide, 40 parts refined paraffin wax and 930 parts of solvent. Cotton swatches were then sprayed with material as described in Example 2 and additional swatches were dipped and treated as described in Example 1. The spray rating tests were conducted and the cotton fabric was found to have a spray rating of 90.

When 30 parts of zirconium octadecoxide was used as the alkoxide in a solution otherwise identical to that above, cotton swatches either dipped therein or sprayed therewith evidenced a spray rating of 70.

Example 5

Example 4 was repeated and the steps and materials were identical to those employed in that example with the exception that only 10 parts of aluminum octadecoxide and 950 parts of naphtha were used. The spray rating test conducted indicated that both the wool and cotton swatches had a spray rating of 100.

Ten parts of zirconium octadecoxide were substituted for the aluminum octadecoxide in the solution immediately above and wool swatches sprayed with the solution bore a rating of 100. Sprayed cotton swatches showed a spray rating of 90.

Example 6

A solution was prepared containing one part of aluminum isopropoxide, 40 parts of polymerized octadecyl vinyl ether and 959 parts of solvent. A number of wool gabardine and cotton swatches were dipped in the material while others were sprayed with the solution described in Example 2. A 100 spray rating was evidenced by both the wool and the cotton fabric.

Where zirconium isopropoxide was the alkoxide used in a solution otherwise similar to that above, wool swatches either dipped or sprayed indicated a rating of 100. Eighty was the spray rating attributed to cotton swatches which were either dipped or sprayed.

Example 7

A solution was prepared with 100 parts of refined paraffin wax, 16.5 parts of aluminum isopropoxide and 883.5 parts of trichlorethylene. The composition was then placed in an aerosol bomb wherein it constituted 60% by weight of the contents of the bomb; the remaining 40% consisting of the propellent, dichlorodifluoromethane. Wool and cotton swatches were sprayed with the solution delivered from the aerosol bomb, and both types of material indicated a spray rating of 100.

16.5 parts of zirconium isopropoxide was the alkoxide employed in a solution otherwise identical to that above and both wool and cotton swatches sprayed with the solution displayed a rating of 100.

Example 8

A solution was prepared with 40 parts of petrolatum having a melting point of from 131° F. to 135° F. and 2 parts of aluminum isopropoxide. Cotton and wool swatches were dipped in and sprayed with the material and were found to have a spray rating of 90. Where two parts of zirconium isopropoxide was substituted for the aluminum alkoxide in the solution above, both wool and cotton swatches, either dipped or sprayed, indicated a rating of 90.

In order to determine the spray rating of the swatches in the examples, after the procedure described in Example 1, the fabric is examined and compared to a series of photographs of water-contacted fabric which constitute a standard chart for rating the fabrics tested. The following ratings indicate the following conditions:

0. Complete wetting of whole of upper and lower surfaces.
50. Complete wetting whole of upper surface.
70. Partial wetting of whole of upper surface.
80. Wetting of upper surface at spray points.
90. Slight random sticking or wetting of upper surface.
100. No sticking or wetting of upper surface.

This test is described in a Federal Standard Stock Catalog, in section IV (part 5) of a supplement to Federal Specification for "Textiles; General Specifications, Test Methods," dated October 8, 1945.

In the examples, as little as .1 per cent (Example 6) to as much as 3 per cent (Example 4) of alkoxide was incorporated. Where more than 3 per cent of the water-repellent agent is em-

ployed, incidental spotting evidences itself upon the fabric treated. Then, too, as shown in Example 4, the greater quantities of the higher alkoxides, such as aluminum or zirconium octadecoxide, render a satisfactory and greatly improved degree of water-repellency but yet not the optimum evidenced by the 100 spray rating in the other examples. Not less than .1 per cent of the water-repelling agent should be used, for if less than that amount be incorporated into the composition, a degree of water-repellency will be rendered which is not greatly improved over that of compositions teaching the application of waxy materials without the addition of the specific alkoxides. The alkoxides, the water-repelling agents, include alkoxides having as little as three carbon atoms (aluminum and zirconium isopropoxide) to alkoxides having as many as eighteen carbon atoms (aluminum and zirconium octadecoxide).

Not less than one per cent nor more than 10 per cent of petroleum wax, petrolatum, or polymerized octadecyl vinyl ether should be included in the solution. Where more than 10 per cent by weight of the composition is one of these materials, the film formed by the solution upon the fabric gives the fabric an undesirable stiffening effect. Where less than 1 per cent by weight of the waxy material is contained in the solution, as shown in Example 3, the degree of water-repellency imparted to the fabric is substantially lowered and practically of no improvement over heretofore disclosed repelling compositions.

It should be appreciated that no vegetable or animal waxes may be utilized, and only the specific waxy materials as set forth above, since it is only these materials which are completely soluble at room temperature which offer a substantial increase in water-repellency when combined with the water-repelling alkoxides. It is mandatory that the waxy material be completely in solution at room temperature and that no "throw out" occur (as would be the case with vegetable and animal waxes), since if such were the condition, the minute solidified particles of the waxy material would evidence themselves upon the fabric treated. However, upon treating fabric with a solution in which any of the specified materials is included as the waxy constituent, within the specific quantity range, no visual trace of the material is observed upon the fabric.

Whereas certain solvents are disclosed in the examples, and volatile liquid, hydrocarbon or halogenated hydrocarbon is operable. The hydrocarbons include benzene, xylene, cymene, dipentene, etc. Examples of operable halogenated hydrocarbons are carbon tetrachloride, trichloroethylene, tetrachloroethylene, etc. It should be appreciated that the operable solvents are critically limited to but these two classes since it has been found that solvents of other classes are either incapable of placing the waxy material or the alkoxide in solution or are deficient in that products employing them as a vehicle often render the treated fabric an undesirable spotting effect. The selected solvent constitutes the major portion by weight of the solution.

Where an alkoxide is employed in a quantity close to the upper limit of the proposed range, and the water-repellent solution is applied to the fabric by dipping it therein, if a sufficient amount of moisture remains in the fabric, spotting of the water-repellent film may occur thereupon. To overcome such a condition, as shown in Example 1, it is advocated that a reactive agent such as octylene glycol be incorporated in the water-repellent solution since this material appears to react with the alkoxide to cause an alcohol exchange, thereby eliminating spotting and causing the aluminum or zirconium compound to be less reactive with water, i. e. rendering it more stable and less susceptible to decomposition.

It has been found that certain resins, such as acrylic resins, pentaerythritol ester of abietic acid, butadiene styrene copolymers, terpene resins, etc., may be incorporated in the solution and, with fabrics of certain texture, aid in the impartment of water-repellency, but in no case may such resins be substituted in lieu of the waxy material.

As illustrated in the examples, methods of applying the solution are not critical, i. e., it has been illustrated that dip, spray and aerosol methods are highly operable and functional.

Obviously, the compositions of the invention are not limited to those which contain any particular number of components but must contain the novel water-repelling alkoxides, and one or more of the specified waxy constituents within the limited ranges.

I claim:

1. A solution for rendering fabric water-repellent, consisting essentially of from 1 to 10% by weight of a material selected from the group consisting of petroleum waxes and polymerized octadecyl vinyl ether from .1% to 3% of an alkoxide selected from the group consisting of aluminum and zirconium alkoxides having from 3 to 18 carbon atoms and a volatile liquid hydrocarbon solvent, said solvent constituting the major portion by weight of said solution.

2. A solution for rendering fabric water-repellent, consisting essentially of from 1 to 10% by weight of petroleum waxes, from .1% to 3% of an alkoxide selected from the group consisting of aluminum and zirconium alkoxides having from 3 to 18 carbon atoms and mixtures thereof, and a volatile liquid hydrocarbon solvent, said solvent constituting the major portion by weight of said solution.

3. A solution for rendering fabric water-repellent, consisting essentially of from 1 to 10% by weight of polymerized octadecyl vinyl ether, from .1% to 3% of an alkoxide selected from the group consisting of aluminum and zirconium alkoxides having from 3 to 18 carbon atoms and a volatile liquid hydrocarbon solvent, said solvent constituting the major portion by weight of said solution.

4. A solution for rendering fabric water-repellent, consisting essentially of from 1 to 10% by weight of a material selected from the group consisting of petroleum waxes and polymerized octadecyl vinyl ether, from .1% to 3% of aluminum isopropoxide, and a volatile liquid hydrocarbon solvent, said solvent constituting the major portion by weight of said solution.

5. A solution for rendering fabric water-repellent, consisting essentially of from 1 to 10% by weight of petroleum waxes, from .1% to 3% of aluminum isopropoxide, and a volatile liquid hydrocarbon solvent, said solvent constituting the major portion by weight of said solution.

6. A solution for rendering fabric water-repellent, consisting essentially of from 1 to 10% by weight of polymerized octadecyl vinyl ether, from .1% to 3% of aluminum isopropoxide, and a volatile liquid hydrocarbon solvent, said solvent

constituting the major portion by weight of said solution.

7. A solution for rendering fabric water-repellent, consisting essentially of from 1 to 10% by weight of petroleum waxes, from .1% to 3% of aluminum isopropoxide, and naphtha serving as a mutual solvent for said petroleum wax and said aluminum isopropoxide, said naphtha constituting the major portion by weight of said solution.

8. A solution for rendering fabric water-repellent, consisting essentially of from 1 to 10% by weight of polymerized octadecyl vinyl ether, from .1% to 3% of aluminum isopropoxide, and naphtha serving as a mutual solvent for said vinyl ether and said aluminum isopropoxide, said naphtha constituting the major portion by weight of said solution.

NORMAN G. MAILANDER.

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