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METHOD OF FINISHING TEXTILES

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This invention relates to textiles having an improved finish and to a method for finishing textiles. More particularly it relates to textiles having a finish which is water repellent and which retains this property upon repeated laundering and to a method for applying this improved finish.

Textiles have been rendered resistant to water by treatment with insoluble soaps, such as aluminum stearate; by treatment with waxes or mixtures of wax and insoluble soap; or by treatment with resins or lacquers in which waxy materials have been incorporated. Water resistance has also been imparted by reacting chemically upon the textile so as to change the inherent nature of the material or by condensing resins upon the textile. Textiles have also been coated with a substantially continuous film to impart water-proofness.

None of these methods have proved completely satisfactory since the finishes imparted have not been very resistant to laundering. The various waxy materials and insoluble soaps are emulsified under the usual conditions of laundering and they tend to be soluble in dry-cleaning solvents. When waxes are incorporated with resins or lacquers they destroy the anchorage of these materials to the fibers and thus such finishes are removed by the scouring action of laundering. The method of reacting upon the fiber to impart water resistance improves somewhat the permanency of such finishes but this method calls for several steps in addition to the usual finishing operations and requires special skill and equipment not available in textile finishing plants. It thus adds unduly to the cost and difficulty of finishing the material. Finishes in which resins and lacquers are incorporated in the textile without the use of waxy materials do not have the water repellency desired and usually are not very resistant to alkaline laundering agents. Coated textiles which repel water by virtue of a substantially continuous coating are impervious to air.

It is an object of this invention to provide a textile finished in such a manner as to render it highly repellent to water. It is a further object of this invention to render textiles water repellent without affecting the permeability of the textiles to air. It is a still further object to render textiles water repellent and in the same operation to obtain a full handle in the textile without harshness and, furthermore, with any degree of stiffness desired.

It is also an object to provide textiles which will retain these desirable properties after laundering without any need for refinishing. It

is also an object to accomplish these results in an expedient manner which may readily be applied with the skill and equipment ordinarily available in textile finishing plants with little or no increase in the cost or time required to finish the textiles.

Now in accordance with this invention, I accomplish these objects by treating a textile with an aqueous emulsion of a composition comprising a higher fatty acid ester of an ethyl ether of cellulose dissolved in a volatile solvent therefor, drying the treated textile and heating the dried material sufficiently to fuse the composition into the fibers. In accordance with this method, penetration into the interstices of the fibers is obtained without coating the yarn or fabric or threads of the fabric in the usual sense of a continuous coating, and substantially without causing adhesion between separate threads. Suitable modifying agents may be incorporated with the essential ingredient of the composition to regulate stiffness and handle of the finished textile.

The product in accordance with this invention consists of a sized textile, the individual fibers of which are in intimate association with a composition comprising a higher fatty acid ester of an ethyl ether of cellulose, with substantially no bonding of thread to thread and with substantially no continuous coating from thread to thread or around the threads. The amount of sizing composition present will ordinarily be within the range of from about 1% to about 5% of the dried, treated textile although beneficial effects are obtained at as low an amount as .2% of the sized textile and, in exceptional cases, the quantity of composition may be as high as 8 to 10% of the sized textile.

The sized textile in accordance with this invention is characterized by being repellent to water. It is further characterized by retaining this property after washing in the usual hot, soap-containing laundering solutions, the finish thus being fast to laundering or permanent as distinguished from finishes not resistant to laundering. It is also resistant to other cleaning solutions. It is further characterized by possessing a substantially unchanged permeability to air and an increased fullness of handle as compared with the original untreated textile.

The particular fatty acid ester of cellulose ethyl ether used in the method and product of this invention is a chemically mixed derivative of cellulose having a sufficient combined degree of substitution to render it soluble in the usual volatile organic solvents. An average of at least

two of the three reactable hydroxyls of each six carbon cellulose unit are substituted and preferably the total substitution is above 2.7. The degree of ethylation of the cellulose may be from about 1.7 to about 2.7, and preferably is from about 2.3 to about 2.5 of the 3 reactive hydroxyls per six carbon units. The degree of esterification of the cellulose by the higher fatty acid will be from about .2 to about 1, preferably from about .3 to about .6 of the 3 reactive hydroxyls per six carbon units. It is to be noted that the higher fatty acid utilized esterifies the cellulose directly, reacting upon hydroxyl groups not affected by the ethylation. The viscosity in an 80-20 toluene-butanol solution may be equivalent to that of any of the types of organic solvent soluble ethyl cellulose commonly used in the lacquer and plastic arts.

The higher fatty acid radical of this cellulose derivative may be any fatty acid radical having 12 or more carbon atoms, for example, stearic, palmitic, oleic, ricinoleic, myristic, lauric, etc.

A high degree of total substitution is preferable. However, it has been found that a degree of substitution by the higher fatty acid above about one tends to impair the permanency of the finish. A relatively low degree of fatty acid esterification with the remainder of the cellulose hydroxyls substantially completely ethylated is productive of unusual resistance to alkali. As a result, alkaline soap solutions have no chemical effect, upon the derivative and furthermore do not remove it by their detergent action from the fibers into which it has been fused.

The ethyl cellulose higher fatty ester may be readily prepared by treating ethyl cellulose of the type soluble in organic solvents with the acid chloride or acid anhydride of the higher fatty acid in solution with the aid of a suitable catalyst. For example, the ethyl cellulose may be dissolved in pyridine, treated with stearyl chloride, precipitated with hot water, washed and dried. Another method of preparation comprises, for example, dissolving the ethyl cellulose in toluene and reacting it with stearic anhydride using paratoluene sulfonic acid as a catalyst, precipitating the product in methanol, washing it with methanol, and drying it.

The two examples following are given to illustrate more fully the preparation of ethyl cellulose higher fatty acid esters which may be utilized in the practice of this invention. All parts are by weight unless otherwise specified.

Example 1

Twenty parts of ethyl cellulose having an ethoxyl content of 47-48% were dissolved in 45 parts of pyridine. Twenty parts of stearyl chloride were then added to this solution and the mixture was agitated for 20 minutes at 110° C. The reaction product was precipitated by the addition of hot water. It was then washed with a hot 0.2% sodium carbonate solution followed by washing with distilled water; finally it was dried for 30 minutes in an oven at 100° C.

Example 2

Ninety-eight parts of ethyl cellulose having an ethoxyl content of 47-48% were dissolved in 350 parts of toluene. Two-hundred and twenty parts of stearic anhydride were added to the solution while heating and agitating. When the mixture became homogeneous, one part of paratoluene sulfonic acid was introduced and the mixture agitated and maintained at a temperature of

110 to 120° C. for about 2 hours. The product obtained was then precipitated with methanol. The precipitate was washed with methanol and then dried at 40° C. in a vacuum oven.

In preparing the emulsion which I use in the practice of this invention, the higher fatty acid ester of the cellulose ethyl ether is dissolved in an organic solvent or solvent mixture which is substantially immiscible with water and which has a moderate or fairly low evaporation rate. The solvent may be, for example, toluene, xylene, high flash naphtha, methyl isobutyl ketone, a hydrogenated naphtha such as Solvesso No. 2, etc., or it may be a solvent mixture such as an 80-20 mixture of xylene and butanol, 80-20 mixture of a hydrogenated naphtha such as Solvesso No. 2 and butanol, etc.

The solution of the sizing composition may contain, in addition to the hereinbefore mentioned cellulose derivative, suitable plasticizers up to about 10% of the non-volatile components, or resins up to about 20% of the non-volatile components. For example, where a limp water repellent finish is desired in the textile, plasticizers, such as hydrogenated methyl abietate, hydrogenated ethyl abietate, triphenyl phosphate, tricresyl phosphate, refined mineral oil, etc., may be used. Where a particularly stiff finish is desired in the textile, resins may be incorporated with the cellulose derivative. For example, resins such as polymerized hydrocarbon resins, cumar resins, polyhydric alcohol esters of rosin or hydrogenated rosin, dammar gum, etc. may be used. It will be appreciated that the resins and plasticizers utilized preferably should be substantially unaffected by dilute alkaline water solutions.

The concentration of non-volatile sizing ingredients will be in the range from about 1% to about 20% of the emulsion, preferably from about 5% to about 10%. The concentration may be widely varied at will according to the textile to be treated and the finish desired therein. The viscosity may be varied to suit the dissolving and emulsifying equipment to be used and the cloth to be finished.

The sizing composition dissolved in the volatile organic solvent is emulsified in water containing emulsifying agents and, if desired, stabilizing agents. The aqueous phase may, for example, contain as emulsifying agents sulfonated oils, such as sulfonated castor oil, sulfonated olive oil, etc.; soaps such as sodium oleate, potassium oleate, ammonium linoleate, sodium resinate, etc.; sodium, potassium and the like salts of higher aliphatic sulfates such as sodium oleyl sulfate, sodium lauryl sulfate, etc.; sodium caseinate, etc. The hereinbefore mentioned solution of the cellulose derivative is agitated with the water phase in which it is to be emulsified in the ratio of, for example, about 2 parts of solution to about 1 part of the aqueous phase. It may then be passed through a colloid mill, homogenizer, or equivalent emulsifying equipment. Usually the emulsion will be prepared, shipped and stored in concentrated form and diluted as used. If desired, the emulsion may be reversed, for example, by addition of acetic acid with agitation, to the water-in-oil type, where a sizing emulsion of high viscosity is required.

In treating the textile by the method in accordance with this invention, the textile may be immersed in the emulsion hereinabove described, preferably after dilution of the emulsion so that the concentration of the non-volatile sizing material will be from about 1% to about 10%. Ex-

cess emulsion may then be removed, for example, by squeezing rolls, and the treated textile dried. Drying may be accomplished, for example, by the usual drying cylinders or by a current of warm air. After the water and volatile solvent have been eliminated, the textile is heated at least to the insipient fusion or softening point of the sizing composition, thereby fusing the composition into the fibers, but at a temperature below that at which appreciable discoloration of the textile takes place. This operation may be performed integrally with the drying operation, for example, on a drying range or it may be performed after the drying operation, for example, in an oven or in a current of heated air, or by a hot iron, or hot rolls, or conveniently by means of a heated calender. The temperature required will vary with the composition of the sizing material; for unmodified ethyl cellulose stearate, for example, a temperature in the range from about 60° C. to about 200° C. preferably from about 100° C. to about 150° C. is suitable. The textile may then be additionally ironed or calendered, if desired.

It will be appreciated that the handle of the finish obtained will vary with the quantity of sizing composition deposited in the textile and with the formation of the sizing composition. The quantity of sizing composition incorporated in the textile may be varied at will by diluting the emulsion.

The examples following are given in further illustration of the method and product of this invention. All parts and proportions mentioned herein are by weight unless otherwise specified.

Example 3

Ethyl cellulose stearate prepared according to Example 2 was dissolved in 80-20 xylol-butanol at a concentration of 30%. This solution was then emulsified in water containing 0.1% triethanolamine and 1.5% potassium oleate at a ratio of 2 parts of solution per part of water. The emulsion obtained was then diluted with 3 parts of water per part of emulsion. A sample of cotton sheeting, 2.85 yards per pound, was impregnated in with the diluted emulsion, and excess emulsion was removed. The treated cloth was dried and heated 15 minutes at 130° C.

Example 4

An emulsion was prepared by dissolving 100 parts of ethyl cellulose stearate in 240 parts of xylene and 60 parts of butanol, agitating this solution with 200 parts of water containing 1.25 parts of potassium oleate and passing the mixture through a colloid mill. The emulsion was then diluted with water so as to contain 8% of ethyl cellulose stearate. A swatch of cotton duck cloth weighing 7 ounces per yard was padded through the dilute emulsion, squeezed through rubber wringer rolls and ironed dry at a temperature of 130° C. The finished fabric was highly repellent to water and possessed a stiff finish fast to repeated laundry washes. The finished fabric was highly pervious to air.

Example 5

One-hundred parts of ethyl cellulose stearate and 15 parts of a dihydro-naphthalene resin having a melting point of about 135° C. were dissolved in 240 parts of xylene and 75 parts of butanol. This solution was agitated with 210 parts of water containing 1.25 parts of sulfonated castor oil (75% grade) and 0.5 part of triethanolamine, and an emulsion formed by passing the mixture through

a colloid mill. The emulsion obtained was diluted with water to a concentration of the non-volatile components of 5%. Lace curtain material was padded through the dilute emulsion, squeezed through rubber wringer rolls, air dried at 120° C. for 4 minutes and ironed for 3 minutes at 120° C. The sized curtain material possessed a stiff crisp finish having excellent water repellent properties. The finish was unaffected by several laundry washes.

Example 6

Seventy-five parts of ethyl cellulose stearate were dissolved in 250 parts of xylene and 50 parts of butanol and the solution agitated with 200 parts of water containing 1.25 parts of sodium lauryl sulfate. An emulsion was formed by passing the mixture through a colloid mill. Three hundred parts by volume of the emulsion were mixed with 600 parts by volume of a direct color dye bath containing 1% Direct Brown RS, Color Index 596. Lace curtain material was padded through the combined dye and finishing bath, squeeze-rolled, dried, and heated. The finish obtained was crisp, water repellent, and fast to laundering. The fastness of the dye-stuff to washing was improved by the presence of the ethyl cellulose stearate.

Example 7

A portion of the concentrated ethyl cellulose stearate emulsion which was used in Example 6 was diluted with water in the ratio of 300 parts by volume of emulsion to 1200 parts by volume of water. Crepe rayon was padded through the diluted emulsion, passed through squeeze rolls, and air dried under tension in a current of air at 105° C. The finished, crepe rayon possessed a spot resistant, water repellent finish. This finish was resistant to cleaning solutions.

Example 8

A cotton warp yarn was sized by passing it through a diluted emulsion similar to that used in Example 7, passing it through rubber squeeze rolls, drying it, and treating it with a current of air at 130° C.

The method in accordance with this invention allows the finishing of textiles to the crisp finishes usually desired on cotton duck, linen, rayon crepe, suiting, dress goods, lace, etc., used for waiters' uniforms, coats, uniforms, dresses, curtains, etc., at the same time imparting permanent water repellency. Furthermore, the degree of stiffness may be modified at the will of the finisher. In addition, the finished material is resistant to soiling and spotting, or if spotted, is usually readily cleaned with a damp cloth. The finishing operation may be combined with the dyeing operation by direct or neutral dye baths by the mere addition of the finishing emulsion to the dye bath.

By the term "textile" used herein and in the appended claims, I mean goods manufactured by weaving such as cotton, silk, linen, rayon, etc. cloth; and also material capable of being woven such as cotton, rayon, woolen, linen, hemp, etc. yarns. The description of the present invention is considered to refer not only to textile fabrics but also to be applicable to textile yarns.

By the term "substitution" used herein and in the appended claims with respect to cellulose derivatives, I mean the average number of cellulose hydroxyls replaced by a given substituent radical per six carbon unit of the cellulose.

It will be understood that the details and examples hereinbefore set forth are illustrative,

and that the invention as broadly described and claimed is in no way limited thereby.

What I claim and desire to protect by Letters Patent is:

1. A method for imparting a permanent water repellent finish to a textile which includes treating the textile with an aqueous emulsion comprising a solution of an ester of a cellulose ethyl ether containing the radical of a fatty acid having at least 12 carbon atoms in a volatile organic solvent so as to leave in the textile, on drying, an amount of impregnating composition which will be insufficient to fill the interstices of the textile or to form a continuous coating thereover, evaporating the water and volatile solvent from the treated textile, and heating the dried textile to a temperature which will fuse the composition deposited therein into its fibers.
2. A method for imparting a permanent water repellent finish to a textile which includes treating the textile with an aqueous emulsion comprising a solution of an ethyl cellulose stearate in a volatile organic solvent so as to leave in the textile, on drying, an amount of impregnating composition which will be insufficient to fill the interstices of the textile or to form a continuous coating thereover, evaporating the water and volatile solvent from the treated textile, and heating the dried textile to a temperature which will fuse the composition deposited therein into its fibers.
3. A method for imparting a permanent water repellent finish to a textile which includes treating the textile with an aqueous emulsion comprising a solution in a volatile organic solvent of an ethyl cellulose stearate having an ethoxyl substitution in the range of about 1.7 to about 2.7 and a stearate substitution in the range of about 0.2 to about 1.0 so as to leave in the textile, on drying, an amount of impregnating composition which will be insufficient to fill the interstices of the textile or to form a continuous coating thereover, evaporating the water and volatile solvent from the treated textile, and heating the dried textile to a temperature which will fuse the composition deposited therein into its fibers.
4. A method for imparting a permanent water repellent finish to a textile which includes treating the textile with an aqueous emulsion of a composition comprising an ethyl cellulose palmitate dissolved in a volatile organic solvent so as to leave in the textile, on drying, an amount of impregnating composition which will be insufficient to fill the interstices of the textile or to form a continuous coating thereover, evaporating the water and volatile solvent from the treated textile, and heating the dried textile to a temperature which will fuse the composition deposited therein into its fibers.
5. A method for imparting a permanent water repellent finish to a textile which includes treating the textile with an aqueous emulsion of a composition comprising a solution in a volatile organic solvent of ethyl cellulose palmitate having an ethoxyl substitution in the range of about 1.7 to about 2.7 and a palmitate substitution in the range of about 0.2 to about 1.0 so as to leave in the textile, on drying, an amount of impregnating composition which will be insufficient to fill the interstices of the textile or to form a continuous coating thereover, evaporating the water and volatile solvent from the treated textile, and heating the dried textile to a temperature which will fuse the composition deposited therein into its fibers.
6. A method for imparting a permanent water repellent finish to a textile which includes treating the textile with an aqueous emulsion of a composition comprising an ethyl cellulose laurate dissolved in a volatile organic solvent so as to leave in the textile, on drying, an amount of impregnating composition which will be insufficient to fill the interstices of the textile or to form a continuous coating thereover, evaporating the water and volatile solvent from the treated textile and heating the dried textile to a temperature which will fuse the composition deposited therein into its fibers.
7. A method for imparting a permanent water repellent finish to a textile which includes treating the textile with an aqueous emulsion of a composition comprising a solution in a volatile organic solvent of ethyl cellulose laurate having an ethoxyl substitution in the range of about 1.7 to about 2.7 and a laurate substitution in the range of about 0.2 to about 1.0 so as to leave in the textile, on drying, an amount of impregnating composition which will be insufficient to fill the interstices of the textile or to form a continuous coating thereover, evaporating the water and volatile solvent from the treated textile, and heating the dried textile to a temperature which will fuse the composition deposited therein into its fibers.
8. A sized textile containing a composition comprising an ester of a cellulose ethyl ether containing the radical of a fatty acid having at least 12 carbon atoms in intimate association with the fibers of the textile, the composition being present in quantity in the range from about 0.2% to about 10% of the sized textile, characterized by high repellency to water, high perviousness to air, and fastness of the finish to laundering.
9. A sized textile containing a composition comprising an ethyl cellulose stearate in intimate association with the fibers of the textile, the composition being present in quantity in the range from about 0.2% to about 10% of the sized textile, said textile being characterized by high permanent repellency to water, high perviousness to air, and fastness of the finish to laundering.
10. A sized textile containing a composition comprising ethyl cellulose stearate having an ethoxyl substitution in the range from about 1.7 to about 2.7 and a stearate substitution in the range of about 0.2 to 1.0, in intimate association with the fibers of the textile, the composition being present in quantity in the range from about 2% to about 10% of the sized textile, said textile being characterized by high permanent repellency to water, high perviousness to air, and fastness of the finish to laundering.
11. A sized textile containing a composition comprising an ethyl cellulose palmitate in intimate association with the fibers of the textile, the composition being present in quantity in the range from about 0.2% to about 10% of the sized textile, said textile being characterized by high permanent repellency to water, high perviousness to air, and fastness of the finish to laundering.
12. A sized textile containing a composition comprising ethyl cellulose palmitate having an ethoxyl substitution in the range from about 1.7 to about 2.7 and a palmitate substitution in the range of about 0.2 to 1.0, in intimate association with the fibers of the textile, the composition being present in quantity in the range from about 2% to about 10% of the sized textile, said textile

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being characterized by high permanent repellency to water, high perviousness to air, and fastness of the finish to laundering.

13. A sized textile containing a composition comprising an ethyl cellulose laurate in intimate association with the fibers of the textile, the composition being present in quantity in the range from about .2% to about 10% of the sized textile, said textile being characterized by high permanent repellency to water, high perviousness to air, and fastness of the finish to laundering.

14. A sized textile containing a composition

comprising ethyl cellulose laurate having an ethoxyl substitution in the range from about 1.7 to about 2.7 and a laurate substitution in the range of about .2 to 1.0, in intimate association with the fibers of the textile, the composition being present in quantity in the range from about .2% to about 10% of the sized textile, said textile being characterized by high permanent repellency to water, high perviousness to air, and fastness of the finish to laundering.

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CERTIFICATE OF CORRECTION.

April 27, 1943.

WILLIAM W. TROWELL.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 4, second column, line 36, claim 8, before "characterized" insert --said textile being--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 15th day of June, A. D. 1943.

(Seal)

Henry Van Arsdale,
Acting Commissioner of Patents.