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PRODUCTION OF WATER-REPELLENT TEXTILES

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This invention relates to the production of improved, water-repellent textiles or other fibrous materials, and more particularly to novel methods for treating such materials to impart permanent water-repellent characteristics thereto.

The treatment of textile materials with emulsions of paraffins, waxes, resins, and the like, to render them water-repellent has already been proposed. However, on subsequent customary laundering, washing or cleaning of the textile with detergents, such as soaps or with solvents, these treating agents are, for the most part, removed and the desired water repellency becomes lost. A real need has thus existed for a water-repellent type of finish or impregnating agent which is useful in treating textile or fabric materials which advantageously will be capable of permanent retention on the fabric to impart permanent water-repellency characteristics thereto.

It is among the objects of this invention to overcome the above and other disadvantages characterizing prior attempts to obtain a water-repellent textile material and to provide a novel type of treating agent and method of rendering textile materials substantially permanently water-repellent. A particular object is to provide a permanent water-repellent finish on fabrics or other textiles which will remain substantially unaffected by the customary laundering or other cleaning treatments to which the same may be subjected during use. Other objects and advantages of the invention will be apparent from the ensuing description of my invention.

These objects are accomplished in this invention which broadly comprises treating textiles or similar fibrous materials with a soluble titanium compound and a long chain (containing from 12 to 20 carbon atoms) fatty acid ester of a polyhydric alcohol (said ester having at least one alcoholic hydroxyl and said polyhydric alcohol being free from other reactive groups) in order to impregnate said textile or fibrous materials with the resulting reaction product or insoluble titanium compound.

In a more specific and preferred embodiment, the invention comprises treating textile materials, such as cotton fabrics, at room or slightly-elevated temperatures, with a combination of a water-soluble titanium salt, such as titanyl sulfate, and a long chain monocarboxylic acid ester of glycerol, such as glycerol monostearate, and then neutralizing, drying and recovering the resulting impregnated, water-repellent material.

To a clearer understanding of the invention, the following specific examples are given. These

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are merely illustrative and are not to be construed as limiting the underlying principles of the invention:

Example I

Khaki-dyed poplin is rendered water-repellent by first impregnating it with glycerol monostearate by treatment in a hot aqueous dispersion of glycerol monostearate for about 10 minutes, followed by treating for one hour at room temperature in a 25% aqueous solution of titanyl sulfate saturated with glycerol monostearate. The treated fabric is then passed through a 2% ammonia solution, and washed thoroughly with water, and dried. The finished fabric, after subjection to a laundering test, exhibits high water-repellency characteristics upon being subjected to a standard spray test, in comparison with a treated control. Controls treated with glycerol monostearate alone, and glycerol monostearate plus sulfuric acid possess no water repellency after laundering. The water repellency of the titanium-treated fabric is retained after washing in alcohol or chloroform.

Example II

Cotton twill fabric is rendered water-repellent by treating for about 5 minutes in a saturated acetone solution of glycerol monostearate, followed after drying by treatment at room temperature for about 30 minutes, in a 17% solution of titanyl sulfate saturated with glycerol monostearate. The treated fabric is then thoroughly washed in cold water to remove excess titanyl sulfate and free sulfuric acid. The finished material possesses excellent water-repellent properties after laundering with soap and warm water.

Example III

Cotton fabric is treated for one hour in a 17% aqueous solution of titanyl sulfate saturated with glycerol monostearate, then passed through a 2% solution of ammonia, and finally washed with water and dried at 100–110° C. The fabric so treated contains about 5% of titanium, shows excellent initial water-repellency, which is retained even after several launderings with soap and hot water, and after washing in alcohol.

Example IV

Example III is duplicated, except that a cotton fabric is treated with 12% aqueous solution of titanyl sulfate saturated with the monolauryl ether of glycerol. The treated fabric is found

to exhibit substantially similar, improved water-repellent properties.

As already stated, the foregoing examples illustrate in detail certain aspects of the invention, but it is to be understood that the invention is not limited thereto. Thus, in lieu of titanyl sulfate, other soluble inorganic or organic compounds or salts of titanium, as well as varying amounts of these compounds, can be employed herein. For example, titanium tetrachloride, titanium bromide, titanium sulfate, titanium nitrate, titanium potassium oxalate, and titanium acetate or lactate, etc., can be used. The TiO_2 concentration of such solutions can range from about 1% to 30%, but preferably ranges from 15–25%.

Although preferably glycerol monostearate is used in combination with a soluble titanium compound to form upon or impregnate a fabric with water-repellent finish, other long chain fatty acid esters of a polyhydric alcohol in which the ester contains at least one alcoholic hydroxyl and the polyhydric alcohol is free from other reactive groups can be employed. Long-chain (containing from 12–20 carbon atoms) monocarboxylic acid esters of glycerol, such as glycerol monostearate or laurate, are especially useful, as are products resulting from the esterification of glycerol with other acids, such as those of myristic, palmitic, stearic, oleic, etc. Again, while the glycerol esters mentioned are preferred for use herein, those obtained from the esterification of other polyhydric alcohols which are free from reactive groups other than the alcoholic hydroxyl groups, such as from ethylene glycol, propylene glycol, pentaerythritol and serbitol, etc., can also be utilized.

Similarly, while especially useful for applying a water-repellent finish to a textile article, such as cotton fabric, the invention can also be applied to other woven materials, including those made of wool, rayon, nylon, etc., or to unwoven fibers, threads or filaments, to coat or impregnate the same. The term "textile," here and in the claims, is generic to all such materials.

The process of coating the fabrics with the contemplated water-repellent agents is preferably carried out at temperatures ranging from about 25° C. to 50° C., and generally may range from 15–100° C., the higher order of temperature advantageously increasing the rate of processing of the fabrics.

As solvents for the preparation of solutions of titanium salts and glycerol esters, water, acetone, alcohol, or other solvent systems determined by the solubility characteristics of the titanium salts and the long-chain organic compounds can be employed.

As noted in the examples, after titanation the textile is passed through an ammoniacal solution to effect neutralization thereof and remove excess residual acid and avoid tendering of the fabric. Such residual acid removal can be effected by extended washing treatment in water or by resorting to other neutralizing agents, such as alkali metal hydroxides or carbonates generally, including potassium or sodium hydroxide or carbonate, ammonium hydroxide, or related materials.

I claim as my invention:

1. A water-repellent textile containing the reaction product of a soluble titanium compound with a long-chain fatty acid ester of a monomeric polyhydric alcohol, said ester containing at least 1 alcoholic hydroxyl and its long-chain fatty acid component having from 12 to 20 carbon atoms.

2. A water-repellent textile containing the reaction product of a water-soluble titanium salt with a long-chain monocarboxylic acid ester of glycerol, said long-chain fatty acid component containing from 12 to 20 carbon atoms.

3. A water-repellent textile containing the reaction product of titanyl sulfate with glycerol monostearate.

4. A water-repellent textile containing the reaction product of titanyl sulfate with glycerol monolaurate.

5. A method for rendering a textile material water-repellent which comprises treating said material with solutions of a soluble titanium compound and a long-chain fatty acid ester of a monomeric polyhydric alcohol, said ester containing at least 1 alcoholic hydroxyl and the long-chain fatty acid component thereof having from 12 to 20 carbon atoms, and then neutralizing, drying and recovering the resulting water-repellent textile.

6. A method for rendering a textile material water-repellent which comprises treating said material at temperatures ranging from 15° C. to 100° C. with a combination of a water-soluble titanium salt and a long-chain monocarboxylic acid ester of glycerol, said long-chain component containing from 12 to 20 carbon atoms, and then neutralizing, washing, drying and recovering the resulting water-repellent textile.

7. A method for rendering a textile fabric water-repellent which comprises treating said fabric at substantially room temperature with a combination of a solution of titanyl sulfate and glycerol monostearate, and then neutralizing, washing and drying the resulting water-repellent fabric.

8. A method for rendering a textile fabric water-repellent which comprises treating said fabric at substantially room temperature with a combination of a solution of titanyl sulfate and glycerol monolaurate, and then neutralizing, washing and drying the resulting water-repellent fabric.

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