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TREATMENT OF WOOL AND SILK

John Gwynant Evans and Charles Edward Salkeld, Blackley, Manchester, England, assignors to Imperial Chemical Industries Limited, a corporation of Great Britain

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7 Claims. (Cl. 8—128)

This application is a continuation-in-part of copending application, Serial No. 113,700, filed by ourselves jointly with Baldwin et al., on December 1, 1936. In the said application, a method has been described for treating textile material, principally cellulosic material, with quaternary compounds of a certain definite structure, whereby to impart to said material a water-repellent finish.

The present application deals with the same general object, but concerns itself primarily with the treatment of animal fibers, more particularly wool and silk fibers.

It is an object of this invention to provide a process for treating wool or silk fibers, in any of their forms, whereby to impart thereto a novel finish characterized primarily by water-repellent properties. Other and further important objects of this invention will appear as the description proceeds.

The term wool in this application includes natural wool which has been chemically treated for the purpose of modifying its dyeing properties or other properties, for example, diminishing its shrinking or felting properties, as in the case of the so-called "chlorinated" wool; and further includes those artificial fibers, made from protein material such as casein, which are known as "artificial wool" or "synthetic wool."

According to the present invention we impregnate the said materials with a solution or dispersion of a quarternary ammonium salt as hereinafter defined, and we then heat the so-impregnated material, preferably after previous drying, to the decomposition temperature of the said salt.

The quaternary ammonium salts to be used according to the invention have the structure

$$R{-}CO{-}N(R'){-}CH_2{-}NX{-}Y$$

wherein R stands for an aliphatic radical containing at least 11 carbon atoms; R' stands for hydrogen or a lower alkyl radical; the group NX stands for the residue of a tertiary base, and may signify a tertiary aliphatic base, or may stand for the residue of a heterocyclic base, such as pyridine or quinoline.

Finally, the symbol Y stands for the monovalent anion of an acid, for instance, of hydrochloric, hydrobromic, nitric, sulphuric, p-toluenesulphonic, oxalic, acetic, formic and the like.

Quaternary ammonium salts corresponding to the above defined structure are, for example, stearamidomethyl-pyridinium chloride, lauramidomethyl-pyridinium - pyridinesulphate, stearomethylamido-methyl-pyridinium chloride, stearamidomethyl - pyridinium pyridine - sulphite, stearamidomethyl-pyridinium pyridine-sulhpate, stearamidomethyl-pyridinium bromide, stearamidomethyl-pyridinium nitrate, oleylamidomethyl-pyridinium chloride. A convenient method of making some of the quarternary salts to be used according to the present invention is described in British Specification No. 471,130. Other suitable methods are the subject of U. S. Patent No. 2,146,392.

Impregnation may conveniently be brought about by padding the material in, for example, an aqueous or organic solvent solution of the quaternary salt. Alternatively the quaternary salt may be applied as a dispersion in any suitable medium. The impregnated material is preferably, but not necessarily, dried prior to the heating which is designed to bring about the decomposition of the quaternary salt. Drying is preferably carried out at a relatively low temperature (to be called for convenience the drying temperature). The heating is at a higher temperature (conveniently called the baking temperature).

The drying temperature is preferably kept low in order to minimize premature decomposition of the quaternary salt. Speed of drying is, however, of importance. The higher drying temperatures (for example, 80° C.) may be used if drying conditions are so arranged that drying, that is evaporation of liquid, takes place rapidly in about 3 minutes. These conditions can be achieved by drying in a brisk current of hot air.

The baking temperature will vary according to the results desired and the quaternary salt to be used. Generally speaking a baking temperature of 90 to 120° C. gives optimum results but the results are in part determined also by the time of heating. The time of heating can be shortened at higher baking temperatures. For instance, if stearamido-methyl-pyridinium chloride is used and the baking is carried out at 120° C., a baking time of at least 3 minutes is desirable, whereas at 150° C., a baking time of 1 minute will suffice.

In some cases the drying and heating steps can conveniently be merged into one operation. That is, the wet, impregnated material may be heated directly to the baking temperature without predrying.

If desired, mildly alkaline agents especially substances having a buffering action, may be added to the impregnating bath. As examples of such agents may be mentioned, borax, hexamethylene

tetramine, magnesium hydroxide, pyridine and aniline. One the other hand acid agents which are known to assist the penetration of wool or silk by aqueous media such as sulphuric, hydrochloric, phosphoric or acetic acids, or sodium dihydrogen phosphate may be added to the impregnating bath.

Treatment of wool and silk according to the process of the present invention imparts novel "finishes" to the said materials. The novel finishes are characterized by one or more of the following properties, enhanced water repellency, soft handle, ease of draping into attractive folds and alteration of dyeing properties. Where the wool or silk has been colored by dyeing or printing prior to treatment there may be also a fixative action on the dyestuffs which have been used for the coloring. The novel finishes are in general resistant to laundering and to dry cleaning.

The invention is illustrated but not limited by the following examples in which the parts are by weight.

Example 1

Silk material dyed to a 2.5% shade with Disulphine Blue A (Color Index No. 714) is passed in a continuous padding operation at 40° C. through a solution containing 5 parts of stearamidomethyl-pyridinium chloride in 1000 parts of water. The material is squeezed and is then dried in a current of warm air at about 50° C. and is finally heated for 10 minutes at 120° C. The material is now water-repellent and possesses a very soft handle. This finish is not readily destroyed by laundering or dry cleaning treatments. In addition the dyeing now possesses an increased resistance to washing.

Example 2

A worsted material is thoroughly wetted out in warm water and is then treated at 45° C. with a solution containing 10 parts of stearamidomethyl-pyridinium pyridine sulphate in 1000 parts of water. The material is hydro-extracted, dried in a current of warm air at 40° C. and then heated for 15 minutes at 120° C. The so-treated material has a water-repellent, soft finish. This finish is not readily destroyed by dry cleaning or laundering treatments.

Example 3

A silk net fabric is padded at room temperature in a solution containing 5 parts of stearamidomethyl-pyridinium chloride in 1000 parts of water. The fabric is squeezed and then heated for 5 minutes at 120° C. The resulting fabric has a soft highly water-repellent finish. This finish is not readily destroyed by laundering or dry cleaning treatments.

Example 4

A silk fabric dyed to a 1.5% shade with Fast Acid Violet A2R (Color Index No. 758) is treated at 49° C. in continuous padding operation with a solution containing 20 parts of stearamidomethyl-pyridinium chloride in 980 parts of water. After squeezing, the wet fabric is heated for 10 minutes in a current of hot air at 120° C. The fabric is now dry and has a water-repellent, soft finish. The finish is not readily destroyed by laundering or dry cleaning treatments. In addition the dyeing possesses an increased fastness to washing treatment.

Example 5

A chlorinated wool delaine material is passed in a continuous padding operation at 35° C. through a solution containing 5 parts of stearamidomethyl-pyridinium chloride in 1000 parts of water. The material is squeezed so that it retains about its own weight of impregnating solution. It is then dried in a current of warm air at about 45° C. and is finally heated for 5 minutes at 120° C. The fabric is now water-repellent and possesses a very pleasing soft handle. In addition, the fabric is characterized by the ease with which it falls into pleasing folds. These novel properties are not readily destroyed by laundering or dry cleaning treatments.

Example 6

Woolen serge is immersed, until it is thoroughly wetted, in an impregnating bath consisting of a solution of 5 parts of stearamidomethyl-pyridinium chloride in 1000 parts of water. The fabric is squeezed, dried below 40° C. and then heated for 20 minutes at 100 to 105° C. A water-repellent fabric having a soft handle is thus obtained. The finish is resistant to dry-cleaning.

Example 7

A woven natural silk fabric is impregnated, dried and heated as in Example 6. The fabric so-obtained has a water-repellent finish and a soft handle. The finish is resistant to dry-cleaning.

Example 8

Woolen serge is thoroughly wetted with a solution of 5 parts of stearamidomethyl-pyridinium sulphite in 1000 parts of water. The impregnated fabric is then squeezed, dried in a current of warm air (30° C.) and then heated for 20 minutes at 105° C. The fabric so obtained has a water-repellent, soft finish. This finish is resistant to dry-cleaning.

It will be understood that the above examples are merely illustrative, and that the details of procedure may be varied within wide limits, without departing from the spirit of this invention. For instance, the temperature of the baking treatment may vary from 90 to 180° C., the duration of the treatment being conveniently adjusted so as to avoid undue injury to the fiber. In practice the time of the baking-treatment may vary from a few seconds to one hour.

Likewise the drying treatment may vary as to time and temperature as already indicated above.

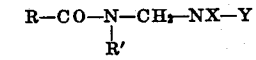
The concentration of the treating agent in the aqueous solution may vary from 0.01% to 2% or even higher if desired. The quantity of the agent applied with respect to the weight of fiber treated may be so chosen by controlling the amount of solution left upon the fiber after the bulk of the liquid has been squeezed out and may be from 0.01% to 2% or more by weight of the fiber.

Many other variations and modifications will be apparent to those skilled in this art.

We claim:

1. The process of treating textile material of animal origin, whereby to impart thereto water repellent surface characteristics, which comprises impregnating the same with an aliphatic acyl-amino-methylene pyridinium halide in which the aliphatic acyl radical contains at least twelve carbon atoms, and heating the impregnated material in the absence of moisture for a period of time sufficient to effect decomposition of the pyridinium compound with liberation of the free base.

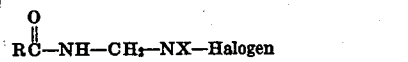
2. The process of treating textile material of animal origin whereby to impart thereto improved surface characteristics, which comprises impregnating the same with a quaternary organic compound of the general formula



wherein R stands for an aliphatic radical containing at least 11 carbon atoms; R' is a member of the group consisting of hydrogen and the lower alkayl radicals; NX stands for the residue of a tertiary base, while Y is the anion of an ionizable acid; and heating the impregnated material at a temperature between 90 and 180° C. in the absence of moisture.

3. A process as in claim 2, the heating being done at a temperature between 105 and 120° C.

4. A process for rendering textile material of animal origin water-repellent which comprises impregnating the material with a quaternary ammonium compound of the formula



in which R stands for an aliphatic radical containing at least eleven carbon atoms and NX for a tertiary amine, and then heating the impregnated material in the absence of moisture at a temperature sufficient to decompose the quaternary ammonium compound with the liberation of the free amine.

5. A process for rendering textile material of animal origin water-repellent which comprises impregnating the material with stearamidomethylene pyridinium chloride, then drying and heating the material at a temperature sufficient to decompose the compound with the liberation of free pyridine.

6. Textile material of animal origin having water-repellant properties and being the product of a process as defined in claim 2.

7. Textile material of animal origin having water-repellant properties and being the product of a process as defined in claim 5.

JOHN GWYNANT EVANS.
CHARLES EDWARD SALKELD.