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PROCESS FOR MAKING WOOL RESISTANT TO FELTING AND SHRINKING

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This invention relates to a process for making wool and wool-containing textile fabrics resistant to felting and shrinking, which comprises treating the wool or fabric with fat-like or resin-like compounds containing epoxide groups and with compounds having a halogenating and/or oxidizing effect.

The treatment of wool with epoxide compounds has previously been disclosed. For example, it is known that wool can be made shrink-proof and at the same time mold-resistant by a treatment with simple low-molecular epoxides which have neither fat-like nor resin-like characteristics. However, such a treatment requires a considerable period of time, and it produces only partially complete effects. It is further known that textile fabrics can be made water-repellent by a treatment with a solution of high-molecular epoxide compounds in organic solvents and subsequent heating of the treated fabric. A resistance against felting and shrinkage is not achieved thereby.

It is an object of the present invention to provide a process for making wool and wool-containing fabrics resistant against felting and shrinking in a single operation.

Other objects and advantages of the present invention will become apparent as the description thereof proceeds.

I have found that wool and wool-containing textile materials can be made resistant against felting and shrinking by a treatment with fat-like or resin-like compounds containing epoxide groups and with compounds having a halogenating and/or oxiding effect.

The process in accordance with the present invention, which can be carried out in a single bath, is characterized over the known processes by a substantial improvement in the resistance against felting and shrinking produced thereby in the wool fiber. At the same time the treatment period is substantially reduced and a considerable saving in chlorine is achieved, so that the wool is treated under mild conditions. Furthermore, the treated wool does not turn yellow and has a very pleasant and soft feel. Finally, the effects achieved thereby are maintained even after repeated launderings of the treated wool and wool-containing fabric.

The epoxide compounds used for the treatment of wool in accordance with the present invention are known compounds and can be produced according to well known methods. Compounds of this type are, for example, the reaction products of epihalogenhydrins with compounds which contain one or more exchangeable hydrogen atoms linked to the molecule through oxygen, sulfur or nitrogen atoms; for example in the form of hydroxyl, sulfhydryl, carboxyl, amino, carbonamide, carbohydrazide or sulfonamide groups. Other epoxide compounds suitable for the present process are those which are formed by a reaction between glycide or its derivatives with the chlorides of monobasic or polybasic organic acids. Furthermore, epoxide compounds are suitable for the present process which are produced by splitting off hydrogen halides from halogenhydrins, or also by a reaction of

peracids with compounds which contain one or more double bonds in the molecule. Finally, also those compounds may be used which are produced by polymerization of unsaturated compounds containing epoxide groups. Some of the above mentioned compounds have a fat-like and some a resin-like character, depending upon the type of starting material used. However, also low molecular epoxides, such as butadienedioxide, are suitable for use in conjunction with the present process, but better effects are produced, as a rule, with the aid of fat-like or resin-like products.

Fat-like epoxides which may be used for the process in accordance with this invention are, for example, the epoxidation products of unsaturated fats, such as soy bean oil, fish train oil or the fatty acids contained in these fats; also the glyceride esters of fatty acids and the like. Suitable resin-like epoxides for the present process are, for example, resin-like glycide esters of succinic acid, adipic acid, sebacic acid, phthalic acid, terephthalic acid or benzenedisulfonic acid, as well as resin-like glycide esters of glycerine, resorcinol, hydroquinone, p,p'-diphenol, dioxydiphenylmethane, diphenylolpropane and the like, and also resin-like polymerization products of methacrylic acid glycide esters, allyl glycide ethers, phthalic acid-allyl-glycide esters and the like. The soft feel of the wool obtained by the process according to the present invention may be further improved by treating the wool with a combination of the resin-like and fat-like epoxide compounds of the type described above.

Suitable compounds having a halogenating and/or oxidizing effect useful for the present process include primarily hypochlorous acid or its salts, which are known to be useful in rendering wool resistant to felting, as well as hypobromites or persulfuric acid.

The epoxide compounds used in accordance with the present invention are preferably applied in combination with the halogenating and/or oxidizing agents in aqueous solution. Since the epoxide compounds are, in most cases, not water-soluble, they are advantageously dispersed in the solution with the aid of suitable emulsifiers. Under some circumstances it is advantageous to first dissolve the epoxide compounds in a suitable organic solvent and thereafter emulsify this solution in the aqueous treatment solution. For this purpose customary solvents may be used, such as acetone, dioxane, tetrahydrofurfuryl alcohol and diacetone alcohol.

The emulsifiers should be resistant against the halogenating or oxidizing agent present in the treatment solution. They may act simultaneously as wetting agents, and in that capacity should be able to produce a wetting effect even in cold solutions and should not foam excessively. For this purpose, such emulsifiers as ethyleneoxide addition products to higher molecular alcohols, as well as similar addition products which are derived from high molecular amines, carboxylic acids, acid amides and the like, are suitable.

The treatment solutions should advantageously contain 0.1 to 1 gram per liter, preferably 0.3 to 0.6 gram per liter of the epoxide compound and about 0.1 to 0.5 gram per liter, preferably 0.3 gram per liter of active chlorine. The impregnation of the wool with this solution is carried out in the usual fashion, for example, in a winch vat or in a yarn-dyeing machine. A treatment period of 5 to 10 minutes is sufficient. Subsequently the wool is rinsed in the usual manner, dechlorinated, if necessary, with such conventional agents as sodium thiosulfate or sodium bisulfite, and finally dried. It is not necessary to heat the treated wool to higher temperatures. The process in accordance with the present invention can be carried out with pure wool as well as with fabrics containing 50% wool (wool-cotton, wool-regenerated wool, wool-rayon, wool-perlon and the like) in the

form of fibers, yarns, ropes, fabrics or also knitted goods.

Aside from the fact that the above-indicated prior art processes involved merely the treatment with epoxide compounds, the present invention can be distinguished over the first of the known processes by the active treatment compound used and over the latter of the known processes by the treatment conditions. Consequently the effects produced by the present process are entirely different.

The following examples will further illustrate my invention and enable others skilled in the art to understand the invention more completely. It is understood, however, that my invention is not limited to the particular conditions and materials recited in these examples.

Example I

50 grams of an epoxide compound (about 5 to 6% epoxide oxygen) produced by a reaction of 1 mol disodiumphthalate and 2 mols epichlorohydrin were dissolved in 200 cc. tetrahydrofurfuryl alcohol, and then 25 grams of the addition product of 5 mols ethyleneoxide to a fatty alcohol mixture wherein the fatty alcohols contained from 12 to 14 carbon atoms were added to this solution as an emulsifier. The epoxide resin solution thus obtained was a clear yellow liquid which was stable over long periods of time.

5 cc. per liter of the above described epoxide resin solution and 12 cc. per liter of glacial acetic acid were stirred into cold water. The emulsion thus obtained was admixed with 0.3 gram per liter of active chlorine in the form of sodium hypochlorite or technical chlorine solution and stirred for a short period of time. Wool in rope form was treated with this emulsion for 5 minutes in an open vat using a solution ratio of 1:30. Subsequently the wool was dechlorinated in the usual fashion with sodium bisulfite, rinsed and dried at 70° C. The wool treated in this manner did not show any reduction in whiteness. Even after repeated laundering the wool remained resistant to felting and shrinking and had a full, soft feel.

In place of the reaction product of disodiumphthalate and epichlorohydrin, a product produced from one mol phthalyl chloride and 2 mols glycide in the presence of an acid-binding agent may be used with equal results.

Example II

45 grams of the epoxide resin used in Example I and 5 grams lauric acid glycide ester were dissolved in 200 cc. dioxane and 25 grams of the emulsifier described in Example I were added to this solution. 700 cc. of glacial acetic acid were then added to the emulsion.

Thereafter 0.3 gram per liter of active chlorine was stirred into cold water in the form of a technical chlorine solution. 10 cc. per liter of the epoxide resin solution were then stirred into the aqueous chlorine solution. An emulsion was formed which was used to impregnate wool in rope form in an open dye vat for 10 minutes using a solution ratio of 1:40. Thereafter the treated wool was dechlorinated with sodium bisulfite in the usual manner, rinsed and dried at 70° C. The treated wool was especially soft and pliable, and fully resistant against felting and shrinking.

Example III

50 grams of the epoxide compound (about 5% epoxide oxygen) produced from 1 mol 2,2-diphenylolpropane and 2 mols epichlorohydrin and 5 grams coconut fatty acid glycide ester were dissolved in 200 cc. acetone and 25 grams of the emulsifier described in Example I were added thereto. Before using this solution for the treatment of wool, 700 cc. of glacial acetic acid were added.

To produce the wool treatment solution, 7 cc. per liter of the epoxide resin solution and then 0.3 gram per liter of active chlorine in the form of sodium hypochlorite were stirred into cold water. Wool or wool-containing fabrics were treated with this solution in a winch vat for 5 to 10 minutes using a solution ratio of 1:40.

Example IV

60 grams of an epoxide resin (about 5 to 6% epoxide oxygen) produced from 1 mol resorcinol and 2 mols epichlorohydrin were dissolved in 200 cc. diacetone alcohol and 25 grams of the emulsifier described in Example I were added to the solution. Before using the solution for treating wool, it was admixed with 700 cc. of glacial acetic acid. To produce the wool treatment solution, 3 grams per liter of persulfuric acid and 6 cc. per liter of the epoxide resin solution were stirred into cold water. A wool fabric was treated with this solution for 10 minutes in a winch vat using a solution ratio of 1:40. Subsequently, the treated wool was thoroughly rinsed and dried at 70° C. The wool fabric was fully resistant against felting and shrinking.

Example V

50 grams of the technical mixture of glycerine mono-, di- and triglycide ether (about 9.5% epoxide oxygen) produced by reacting 3 mols epichlorohydrin with 1 mol glycerine and subsequent release of hydrochloric acid, were dissolved in 200 cc. acetone and 25 grams of the addition product of 6 mols ethyleneoxide to a fatty acid mixture containing carbon chains of 12 to 18 carbon atoms were addedd to the solution.

To produce the wool treatment solution 3 cc. per liter of the epoxide resin solution, 7 cc. per liter of glacial acetic acid and 0.3 gram per liter of active chlorine in the form of a technical chlorine solution were stirred into cold water. Wool in rope form was treated in this solution in an open dye vat for 5 to 10 minutes using a solution ratio of 1:40. Subsequently, the treated wool was dechlorinated with sodium bisulfite in the usual manner, rinsed and dried at 70° C. The finished wool was fully resistant against felting and shrinking.

Example VI

40 grams of the reaction product of adipic acid and epichlorohydrin (about 2 to 3% epoxide oxygen) were dissolved in 200 cc. diacetone alcohol, and 25 grams of the emulsifier described in Example I were added thereto. Before use, this solution was admixed with 700 cc. glacial acetic acid.

To produce the wool treatment solution 8 cc. per liter of the epoxide resin solution and 0.3 gram per liter of active chlorine in the form of a techinal chlorine solution were stirred into cold water. Wool fabric was treated with this solution for 10 minutes in a winch vat using a solution ratio of 1:40. Subsequently the treated wool was dechlorinated, rinsed and dried as above described. The finished wool was fully resistant against felting and shrinking.

Example VII

A polyester was produced from 3 mols adipic acid and 2 mols butanediol. The potassium salt of this polyester was then reacted with epichlorohydrin. 50 grams of this epoxide compound (about 2.7% epoxide oxygen) were dissolved in 200 cc. tetrahydrofurfuryl alcohol. Before use this solution was admixed with 700 cc. glacial acetic acid. For the production of the wool treatment solution 10 cc. per liter of the epoxide resin solution and 0.3 gram per liter active chlorine in the form of a technical chlorine solution were stirred into cold water. Wool in rope form was treated with this solution in an open dye vat for 10 minutes using a solution ratio of about 1:30. Thereafter the treated wool was dechlorinated, rinsed and dried at 70° C. The finished wool fabric was fully resistant against felting and shrinking.

Example VIII

50 grams of a polymethacrylic acid glycide ester (about 9 to 10% epoxide oxygen) were dissolved in 200

cc. acetone and 25 grams of the emulsifier described in Example I were added thereto. To produce the wool treatment solution 5 cc. per liter of the epoxide resin solution, 12 cc. per liter glacial acetic acid and 0.3 gram per liter of active chlorine in the form of sodium hypochlorite were stirred into cold water. Wool fabric in rope form was treated with this solution in an open dye vat for 5 minutes using a solution ratio of 1:30. Thereafter the treated fabric was dechlorinated, rinsed and dried at 70° C. The finished wool fabric was fully resistant against felting and shrinking.

Example IX

10 grams of an epoxidized soy bean oil (about 3.5% epoxide oxygen) were admixed with 10 cc. diacetone alcohol, 75 cc. glacial acetic acid and 5 grams of the emulsifier described in Example I. To produce the wool treatment solution, 5 cc. per liter of this epoxide resin solution were stirred into cold water and 0.3 gram per liter of active chlorine (=3 cc. chlorine solution with an active chlorine content of 100 grams per liter) were added to the emulsion formed thereby. Wool fabrics were treated with this solution for 5 to 10 minutes and subsequently dechlorinated, rinsed and dried as previously described. The finished fabrics were fully resistant against felting and shrinking.

If in place of the epoxidized soy bean oil, an epoxide compound produced by the same process from soy bean fatty acid (about 2.6% epoxide oxygen) or fish train oil (about 4.8% epoxide oxygen) was used, substantially the same favorable effects were achieved.

Example X

A solution was produced from 50 grams of an epoxide soy bean oil (about 3.5% epoxide oxygen), 25 grams of the epoxide resin used in Example I, 75 cc. diacetone alcohol, 360 cc. glacial acetic acid and 25 grams of the emulsifier described in Example I. To produce the wool treatment solution, 5 cc. per liter of this solution and 0.3 gram per liter active chlorine in the form of a technical chlorine solution were stirred into cold water. Wool fabrics and wool-containing fabrics were treated in this solution for 5 to 10 minutes and were subsequently rinsed, dechlorinated and dried. The wool and wool-containing fabrics were fully resistant against felting and shrinking.

While I have illustrated various specific embodiments of my invention, it will be apparent to persons skilled in the art that my invention is not limited to these embodiments and that various changes and modifications can be made without departing from the spirit of the invention or the scope of the appended claims.

I claim:

1. The method of simultaneously felt- and shrink-proofing wool, which comprises immersing said wool in a single aqueous bath containing essentially between 0.1 and 1.0 gm./liter of a polymerized epoxide compound having an epoxide oxygen content from 2 to 10%, said epoxide compound selected from the group consisting of carboxylic acid epoxy-alkyl esters, epoxy-alkyl ethers and epoxidized unsaturated fatty oils, an inert solvent selected from the group consisting of tetrahydrofurfuryl alcohol, dioxane, acetone and diacetone alcohol, an emulsifier selected from the group consisting of ethylene oxide addition products of fatty alcohols with 12 to 14 carbon atoms and ethylene oxide addition products of fatty acids with 12 to 18 carbon atoms, and a compound capable of releasing from 0.1 to 0.5 gm./liter of an element selected from the group consisting of chlorine and oxygen in aqueous solution, said compound selected from the group consisting of alkali metal hypochlorites, hypochlorous acid and persulfuric acid, allowing said wool to remain in said aqueous bath at substantially room temperature for a sufficient amount of time ranging from about 5 to about 10 minutes to become thoroughly impregnated with said aqueous bath, removing the wool from the aqueous bath and drying the impregnated wool at a temperature of about 70° C.

2. The method of simultaneously felt- and shrink-proofing wool, which comprises immersing said wool in a single aqueous bath containing essentially 0.1 to 1.0 gm./liter of a polymerized dicarboxylic acid epoxy-alkyl ester having an epoxide oxygen content of 2 to 10%, an inert solvent selected from the group consisting of tetrahydrofurfuryl alcohol, dioxane, acetone and diacetone alcohol, an emulsifier selected from the group consisting of ethylene oxide addition products of fatty alcohols with 12 to 14 carbon atoms and ethylene oxide addition products of fatty acids with 12 to 18 carbon atoms, and from 0.1 to 0.5 gm./liter active chlorine, allowing said wool to remain in said aqueous bath at substantially room temperature for a sufficient amount of time ranging from about 5 to about 10 minutes to become thoroughly impregnated with said aqueous bath, removing the impregnated wool from the aqueous bath, dechlorinating the impregnated wool with a compound selected from the group consisting of sodium bisulfite and sodium thiosulfate, and drying the wool at a temperature of about 70° C.

3. The method of simultaneously felt- and shrink-proofing wool, which comprises immersing said wool in a single aqueous bath containing essentially 0.1 to 1.0 gm./liter of a polymerized epoxy-alkyl ether having an epoxide oxygen content of 2 to 10%, an inert solvent selected from the group consisting of tetrahydrofurfuryl alcohol, dioxane, acetone and diacetone alcohol, an emulsifier selected from the group consisting of ethylene oxide addition products of fatty alcohols with 12 to 14 carbon atoms and ethylene oxide addition products of fatty acids with 12 to 18 carbon atoms, and from 0.1 to 0.5 gm./liter active chlorine, allowing said wool to remain in said aqueous bath at substantially room temperature for a sufficient amount of time ranging from about 5 to about 10 minutes to become thoroughly impregnated with said aqueous bath, removing the impregnated wool from the aqueous bath, dechlorinating the impregnated wool with a compound selected from the group consisting of sodium bisulfite and sodium thiosulfate, and drying the wool at a temperature of about 70° C.

4. The method of simultaneously felt- and shrink-proofing wool, which comprises immersing said wool in a single aqueous bath containing essentially 0.1 to 1.0 gm./liter of a polymerized epoxidized unsaturated fatty oil having an epoxide oxygen content of 2 to 10%, an inert solvent selected from the group consisting of tetrahydrofurfuryl alcohol, dioxane, acetone and diacetone alcohol, an emulsifier selected from the group consisting of ethylene oxide addition products of fatty alcohols with 12 to 14 carbon atoms and ethylene oxide addition products of fatty acids with 12 to 18 carbon atoms, and from 0.1 to 0.5 gm./liter active chlorine, allowing said wool to remain in said aqueous bath at substantially room temperature for a sufficient amount of time ranging from about 5 to about 10 minutes to become thoroughly impregnated with said aqueous bath, removing the impregnated wool from the aqueous bath, dechlorinating the impregnated wool with a compound selected from the group consisting of sodium bisulfite and sodium thiosulfate, and drying the wool at a temperature of about 70° C.

5. The method of simultaneously felt- and shrink-proofing wool, which comprises treating said wool in a single aqueous bath comprising between 0.1 and 1.0 gm./liter of a polymerized epoxide compound selected from the group consisting of carboxylic acid epoxy-alkyl esters, epoxy alkyl ethers and epoxidized unsaturated fatty oils and a compound capable of releasing an element selected from the group consisting of chlorine

and oxygen in aqueous solution, said compound being present in sufficient amount to release from 0.1 to 0.5 gm./liter of said element, allowing said wool to remain in said aqueous bath at a substantially constant room temperature for a period of time ranging from about 5 to about 10 minutes, to cause impregnation of said wool with said solution.

6. The method of simultaneously felt- and shrink-proofing wool, which comprises immersing said wool in a single aqueous bath comprising between 0.1 and 1.0 gm./liter of a polymerized epoxide compound selected from the group consisting of carboxylic acid epoxy-alkyl esters, epoxy-alkyl ethers and epoxidized unsaturated fatty oils and a compound capable of releasing an element selected from the group consisting of chlorine and oxygen in aqueous solution, said compound being present in sufficient amount to release from 0.1 to 0.5 gm./liter of said element, allowing said wool to remain in said aqueous bath at a substantially constant room temperature for a period of time ranging from about 5 to about 10 minutes to cause impregnation of said wool with said solution, removing said wool from said bath and drying said impregnated wool.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 2,926,064 February 23, 1960

Alfred Rapp

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 4, line 28, for "addedd" read -- added --; column 5, lines 34 and 35, for "epoxide" read -- epoxidized --.

Signed and sealed this 30th day of August 1960.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

ROBERT C. WATSON
Commissioner of Patents