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3,567,499

TEXTILE MATERIALS AND THE PROCESS FOR FINISHING THE SAME

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8 Claims

ABSTRACT OF THE DISCLOSURE

Textile materials are finished by treatment with an aqueous bath which contains a polyurethane prepolymer having free isocyanate groups which in turn has been obtained from an organic compound having active hydrogen containing groups and an organic polyisocyanate, and an organo polysiloxane having a molecular weight of to about 25,000 and containing at least two active hydrogen containing groups. The organo polysiloxane is reactive with the isocyanato prepolymer on the textile substrate in order to give the textile material a full pleasantly soft, supple hand and reduce susceptibility to soiling. One may use polymers or copolymers prepared from vinyl or divinyl monomers in the aqueous bath along with the other components.

This invention relates to textile materials and the process for finishing the same. More particularly, it relates to textile materials treated to improve their hand and softness. It has been proposed heretofore to prepare an aqueous bath for the treatment of textile materials which employs a vinyl polymer and a prepolymer having free-NCO groups. It has also been proposed heretofore to prepare the prepolymer in a first step by reacting an hydroxyl organo siloxane with an organic polyisocyanate and then prepare an aqueous liquor thereof together with a vinyl polymer. These aqueous liquors have then been used heretofore to treat textile materials, however, the resulting treated textile material, whether of natural or synthetic origin does not have an entirely desirable hand. In other words, it is not as full, pleasant, soft and supple as it should be. Furthermore, the ability of these treated materials to resist dirt is not as good as is to be desired.

It is an object of this invention to provide improved textile fabrics. A further object of his invention is to provide textiles having an improved finish, an improved hand, a more supple feel and which have reduced susceptibility to soiling. Another object of this invention is to provide an improved method of treating textile materials. Still another object of this invention is to provide a method of finishing textiles and the finished textiles which may be based on cotton, cellulose, wool, silk, polyamides, polyurethane, polyesters, polycarbonate, polyacrylonitrile, polypropylene and the like.

The foregoing objects and others which will become apparent from the following description are accomplished, generally speaking by providing textile materials which have been finished by treating them with an aqueous bath which contains a reaction product having free isocyanato groups which has been prepared from an organic compound having a molecular weight of about 500 to about 10,000 and which contains at least two active hydrogen containing groups as determined by the Zerewitinoff method and an organic polyisocyanate, in conjunction with an organo polysiloxane having a molecular weight of up to about 25,000 and which contains at least

two active hydrogen containing groups as determined by the Zerewitinoff method. This aqueous bath is used to treat the textile material which may then be dried and subjected to an after treatment with water in order to accelerate the curing of the polyurethane material in conjunction with the organic polysiloxane on the textile substrate. However, it is to be pointed out that the crux of this invention is the use of and reaction of the hydroxy organo polysiloxane with the isocyanato prepolymer in the aqueous bath on the textile substrate since this gives the textile materials an improved hand, better resistance to soiling, and a softer more supple feel than was heretofore known with textiles treated with organo polysiloxane isocyanato prepolymers. The invention therefore could be briefly summarized as follows: the process involves treating textile materials with aqueous baths which contain:

(a) Reaction products containing isocyanate groups, which reaction products are prepared from organic compounds of molecular weight about 500 to 10,000 containing at least two active hydrogen atoms and organic polyisocyanates, and

(b) Organopolysiloxanes of molecular weight up to 25,000 containing at least two active hydrogen atoms and, if desired,

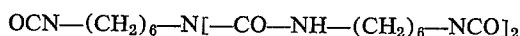
(c) Polymers or copolymers prepared from vinyl or divinyl monomers.

Examples of organic compounds containing at least two active hydrogen containing groups and having a molecular weight of 500 to 10,000 and which are suitable for preparing the reaction products containing isocyanate groups which, to be used according to the invention, are as follows: Polyalkylene glycols such as polyethylene, polypropylene, polybutylene or polyhexylene glycols or copolymers or graft polymers of the alkylene oxides on which these polyglycols are based and polythioetherglycols which are obtainable by condensation of thiodiglycol with itself or with polyalcohols that are free from sulphur. Polyesters which are obtained by the condensation of dicarboxylic acids with polyalcohols or by condensation of hydroxycarboxylic acids are also suitable as well as polyester amides such as are described, for example, in U.S. patent specification No. 2,424,883, and polyacetals such as are described, for example, in U.S. patent specification No. 3,021,289.

As compounds containing at least two active hydrogen atoms those having two or more free hydroxyl groups are preferably used, but those having free carboxyl, mercapto or amino groups are also suitable. The molecular weights of these compounds may be up to 10,000 although the preferred range lies between 750 and 5,000.

In addition to the high molecular weight compounds containing active hydrogen atoms, minor quantities of low molecular weight compounds such as diols, for example, 1,4-butanediol, ethylene glycol, diethylene glycol and the like which have a modifying effect on the properties of the end products may also be used.

Any suitable organic polyisocyanate may be used for the preparation of the reaction products and are preferably aliphatic or cycloaliphatic diisocyanates. Examples of these include tetramethylene diisocyanate, hexamethylene diisocyanate, 1,4-cyclohexane diisocyanate, 4,4'-dicyclohexylmethane diisocyanate, 2,4- and 2,6-hexahydrotoluylene diisocyanate and the like. Aromatic diisocyanates such as p-phenylene diisocyanate and 2,4- or 2,6-toluylene diisocyanate as well as triisocyanates, such as the reaction product of the formula



obtainable by reaction of 3 mols of hexamethylene diisocyanate and 1 mol of water are also suitable. The prepara-

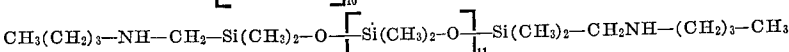
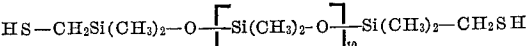
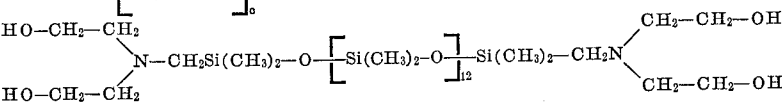
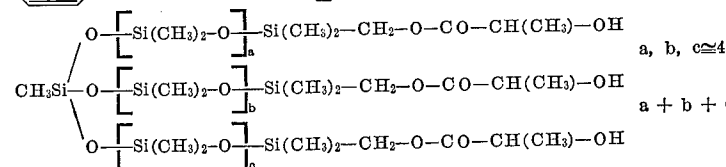
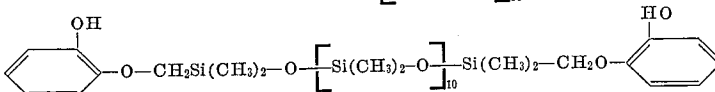
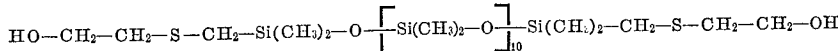
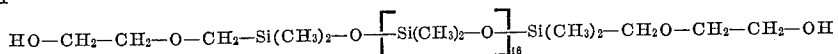
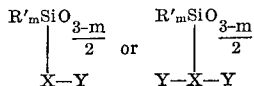
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tion of the reaction products containing isocyanate groups may be performed by reacting the compounds which carry at least two active hydrogen atoms with a stoichiometric excess of polyisocyanate at elevated temperatures. The reaction products which contain isocyanate groups can also be utilized in the form of the known bisulphite addition products.

The organopolysiloxanes having two active hydrogen atoms that are to be used according to the invention include in particular compounds of the general formula

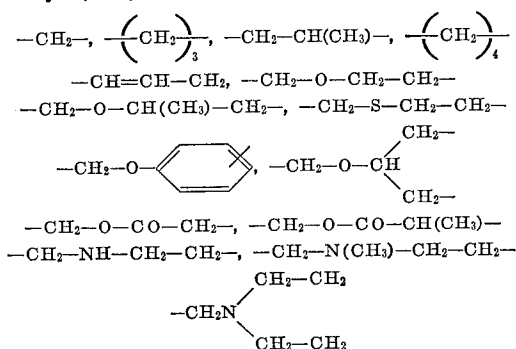


in which n is greater than 1 and not more than 3 and in which each substituent R is one of the radicals R' , $-X-Y$ or $Y-X-Y$, where R' represents an aliphatic, cycloaliphatic or aromatic hydrocarbon radical which may, if desired, be substituted by an inert substituent, X represents an aliphatic or araliphatic bivalent or trivalent radical which may contain an ether, thioether, ester or amino group, and Y represents a hydroxyl, sulphydryl, carboxyl or carbonamide group or a secondary amino group, with the proviso that at least two siloxane units are of the formulae:



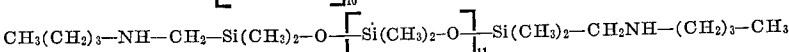
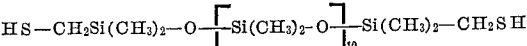
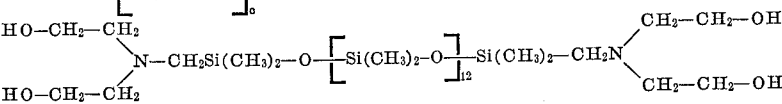
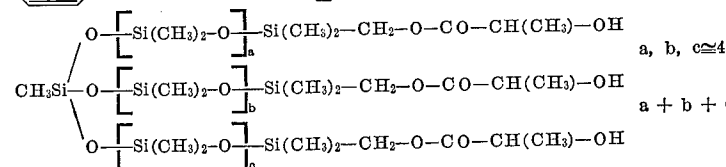
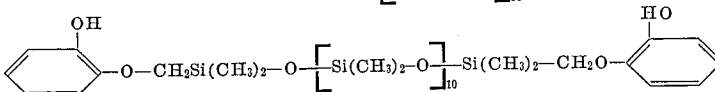
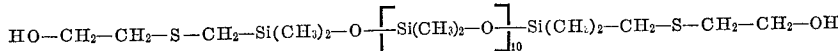
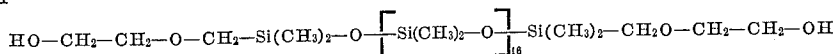
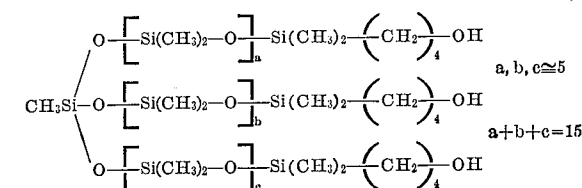
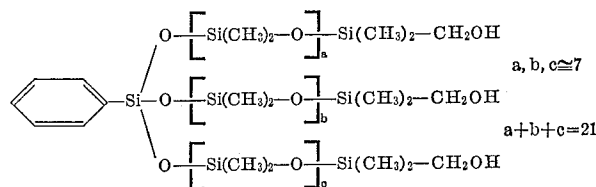
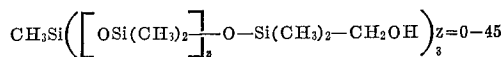
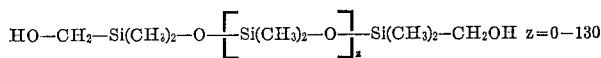
in which R' , X and Y have the meanings given above and m is 1 or 2, and the substituents R in the remaining siloxane units are exclusively the radicals R' .

The following are examples of the hydrocarbon radical R' : methyl, ethyl, isopropyl, tertiary butyl, octadecyl, allyl, chloromethyl, gamma-trifluoropropyl, cyclohexyl, phenyl and tolyl radicals. Those polysiloxanes of the above formula in which R' represents a methyl or phenyl radical are preferred. Bivalent or trivalent radicals, denoted by X , are, for example, the following:



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The following compounds are mentioned by way of example:



Polysiloxanes of this type are known; they have been described, for example, in U.S. patent specifications Nos. 3,297,735, 3,311,651, 3,324,161 and 3,345,393, in British patent specifications Nos. 995,394 and 1,063,085, in German patent specification No. 1,199,772 and in German published specification No. 1,196,869.

Minor amounts of monofunctional organopolysiloxanes may also be used.

The organopolysiloxanes described above can be used as starting materials for the reaction products containing isocyanate groups which are to be used according to the invention.

Examples of vinyl and divinyl monomers on which the polymers or copolymers to be used according to the invention may be based are as follows: ethylene, propylene, vinyl chloride, vinyl acetate, vinyl ethyl ether, styrene or divinylbenzene, butadiene, isoprene or chloroprene and α,β -unsaturated carboxylic acids such as acrylic acid and methacrylic acid as well as their nitriles, esters and amides. Polymers which contain groups capable of reacting with isocyanates are generally to be preferred; these include, for example, polymers or copolymers prepared from acrylic acid, methacrylic acid, their hydroxyalkyl esters or amides, as well as the copolymers which

can be obtained when N-methylolacrylamide, N-methylolmethacrylamide or their derivatives prepared by reacting them with alcohols containing at least one further functional group are copolymerized with other olefinically unsaturated compounds, e.g. by the process according to U.S. patent specification No. 3,243,399.

The proportion of organopolysiloxanes containing at least two active hydrogen atoms may be 1 to 20 equivalents percent, based on the reaction products containing isocyanate groups, provided that each isocyanate group is capable of reacting with one of the active hydrogen atoms of the organopolysiloxanes. The ratio usually lies between 3 and 7 equivalents percent.

The ratio by weight between the reaction products which contain isocyanate groups and the polymers or copolymers which may be used is preferably between 0.25:1 and 4:1. The amount of reaction products containing isocyanate groups, organopolysiloxanes and, where present polymers or copolymers contained in the aqueous bath may also vary within wide limits, for example somewhere between 20 and 300 grams/liter. The most suitable concentration in the bath and the appropriate proportions of the individual components to each other can easily be determined from case to case by preliminary tests.

The impregnating baths can be prepared by emulsifying the reaction products which carry isocyanate groups and the organopolysiloxanes which have at least two active hydrogen atoms and which have been adjusted, if necessary, to a low viscosity by the addition of inert solvents that are immiscible with water, such as benzene, toluene, ethyl acetate, petrol or chlorinated hydrocarbons, by means of a high speed stirrer in water in which suitable emulsifiers such as alkylphenolpolyglycol ethers, commercial paraffin sulphonates, alkyl benzene sulphonates or the alkali salts of acid sulphur acid esters of higher aliphatic alcohols are dissolved in quantities of 0.5 to 10% by weight. In this process, two emulsions, each of which contains one of the components, may first be prepared separately and then combined, if desired with the addition of the polymers or copolymers in the form of their aqueous dispersions. Alternatively, both components may be stirred successively into the same aqueous emulsifier solution by means of a high speed stirrer, and the polymer or copolymer emulsion may then be added to this emulsion. A third method of preparation is possible with the use of polymers or copolymers. In this method, a stable emulsion is first prepared from the organopolysiloxane and the polymer or copolymer emulsions, and this is then mixed with the emulsion of isocyanate group-containing reaction products, which last mentioned emulsion is prepared shortly before use. If desired the baths may then be diluted with water.

The treatment of the textile materials with the aqueous baths to be used according to the invention may be performed by steeping the textile materials in the baths and then squeezing them out or centrifuging them to reduce the weight increase to about 40 to 150%, preferably 70 to 100%, and then drying. Alternatively the aqueous bath may be sprayed on to the textile material which is then dried. In many cases, it is advisable to store the materials for a few days at room temperature after the treatment. The process of storing the finished textile materials for several days, a process which is to be recommended especially in cases where less highly reactive, aliphatic isocyanates are used, can be shortened or even avoided by accelerating the reaction between the isocyanate groups and the compounds which contain active hydrogen atoms. For this purpose, 0.1 to 10 grams/liter of water-soluble salts of metals of sub-Group I, II or VIII of the Periodic System of elements with lower aliphatic carboxylic acids are added to the aqueous bath. The textile materials are impregnated and dried and this operation may be followed by a 3 to 30 minutes after-treatment in water at 10 to 100° C. which water may contain surface-active substances, 1 to 5 grams of hydrogen peroxide (30%) or

0.1 to 1 g. of catalysts which accelerate the isocyanate reaction with compounds containing active hydrogen atoms being added per liter of water at temperatures below 60° C. The after-treatment may also be performed by treating the impregnated and dried textile materials with steam at 100 to 110° C. for ½ to 10 minutes.

Zinc salts of lower carboxylic acids are particularly suitable salts to add to the bath. Suitable catalysts for the after treatment are, for example, tin compounds such as dibutyl tin dilaurate or dimaleate, aqueous solutions of stannous chloride stabilized with glycollic acid, tertiary amines such as triethylamine, dimethylcyclohexylamine, triethylenediamine and N-ethylmorpholine or the acetyl acetates of nickel, cobalt or iron.

The process according to the invention makes it possible to obtain excellent finishes on textile materials of natural or synthetic origin, for example textile materials of cotton, regenerated cellulose, wool or silk as well as of polyamide, polyurethane, polyester, polycarbonate, polyacrylonitrile or polypropylene fibers, especially from the point of view of their crease resistance, abrasion resistance and dimensional stability. In addition, the treated textile materials are distinguished by a full, pleasant, soft and supple handle and an increased dirt repellency. The treated textiles are useful to make clothing such as trousers, coats and the like.

The invention is further illustrated by the following examples in which parts are by weight unless otherwise specified.

EXAMPLE 1

A blended fabric of about 55% polyacrylonitrile fibers and about 45% wool was treated with an aqueous bath which contained, per liter, about 20 parts of an emulsion of a reaction product containing isocyanate groups, about 15 parts of the mixture of an emulsion of an organopolysiloxane with the aqueous dispersion of a copolymer and about 0.3 part of zinc acetate.

The blended fabric of polyacrylonitrile fibers and wool was squeezed out to a bath uptake of about 90%, dried for about 10 minutes at 110° C. and then treated for up to about 1 minute with steam at 102° C. The fabric, treated according to the invention has a soft, smooth handle and the following advantages compared to an untreated fabric:

	Dry creasing angle		Wet creasing angle	
	Warp	Weft	Warp	Weft
Untreated fabric.....	135°	130°	125°	128°
Fabric treated according to the invention.....	160°	161°	145°	142°
	Percent shrinkage after 5 machine washes at 30° C.			
	Warp		Weft	
Untreated fabric.....	5.3		4.2	
Fabric treated according to the invention...	0.9		0.6	
	Abrasion resistance (repenning)			
Untreated fabric, revolutions.....	410			
Fabric treated according to the invention, revolutions.....	520			

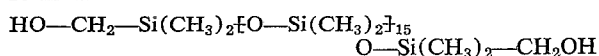
The emulsion of the reaction product containing isocyanate groups was prepared as follows:

About 3,000 parts of a polypropylene glycol which had a molecular weight of about 2000, an OH number of about 55.5 and an acid number of about 0.5 was heated together with about 535 parts of hexamethylene diisocyanate for about 2 hours at about 110° C. and then for about 1½ hours at about 130 to about 140° C. An emulsion was then prepared from about 500 parts of the resulting reaction product which had a free isocyanate group content of about 3.9% by weight with about 100 parts of ethyl acetate and about 400 parts of water with the addition of about 4 parts of a commercial surface-active paraffin sulphonate.

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The mixture of the emulsion of an organopolysiloxane with the dispersion of a copolymer was prepared as follows:

About 400 parts of an organopolysiloxane of the formula



was emulsified with about 596 parts of water which contained about 4 parts of the sodium salt of an alkyl sulphionate.

About 50 parts of this emulsion were mixed with about 1000 parts of the about 40% aqueous dispersion of a copolymer of about 60 parts of butyl acrylate, about 30 parts of styrene and about 10 parts of acrylamide.

EXAMPLE 2

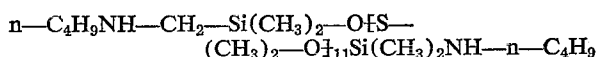
A cotton fabric was treated with an aqueous bath which contained, per liter, about 80 parts of an emulsion of the isocyanate group-containing reaction product described in Example 1, about 60 parts of the mixture of an emulsion of an organopolysiloxane with the aqueous dispersion of a copolymer, about 60 parts of dimethylolethylene urea, about 0.5 parts of zinc acetate and about 10 parts of magnesium chloride.

The fabric was squeezed out to reduce the bath uptake to about 80%, dried at about 100° C. and then after-heated for about 5 minutes at about 150° C. The cotton fabric treated according to the invention has a smooth, soft handle and a high wet creasing angle in addition to good abrasion resistance. The effects are largely resistant to washing.

	Dry creasing angle		Wet creasing angle	
	Warp	Weft	Warp	Weft
Untreated fabric.....	60*	58°	70°	68*
Fabric treated according to the invention.....	136°	141*	140°	145°
Fabric treated with 60 g. of dimethylolethylene urea per liter.....	142°	150°	115°	121°
Abrasion resistance (repenning)				
Untreated fabric, revolutions.....	190			
Fabric treated with 100 g. of dimethylolethylene urea per liter, revolutions.....	160			
Fabric treated according to the invention, revolutions.....	290			

The mixture of the emulsion of an organopolysiloxane with the dispersion of a copolymer was prepared as follows:

About 400 parts of an organopolysiloxane of the formula



were emulsified with about 596 parts of water which contained about 4 parts of a commercial surface-active paraffin sulphionate. About 50 parts of this emulsion were mixed with about 100 parts of the about 40% aqueous dispersion of a copolymer of about 60 parts of butyl acrylate about 30 parts of styrene and about 10 parts of acrylamide.

EXAMPLE 3

A rayon fabric was treated with an aqueous bath which contained, per liter, about 100 parts of an emulsion of the isocyanate group-containing reaction product described in Example 1, about 100 parts of the mixture, described in Example 2, of the emulsion of an organopolysiloxane and the aqueous dispersion of a copolymer, about 70 parts of dimethylolethylene urea, about 0.5 part of zinc acetate and about 15 parts of magnesium chloride. The fabric was squeezed out to a bath uptake of about 100%, dried at about 100° C. and then heated afterwards at 150° C. for 5 minutes. The rayon fabric treated according to the invention has a full, smooth wool-like handle, good abrasion

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resistance and high wet creasing angle. The effects are largely resistant to washing.

	Dry creasing angle		Wet creasing angle	
	Warp	Weft	Warp	Weft
5 Untreated fabric.....	102°	105°	65°	70°
10 Fabric treated according to the invention, after 5 boiling washes in a washing machine.....	140°	151°	142°	148°
15 Fabric treated with 100 g. of dimethylolethylene urea per liter, after 5 boiling washes in a washing machine.....	140°	152°	105°	102°
Abrasion resistance (repenning)				
15 Untreated fabric, revolutions.....	200			
Fabric treated according to the invention, revolutions.....	280			
Fabric treated with 100 g. of dimethylolethylene urea per liter revolutions.....	150			

EXAMPLE 4

20 A fabric of polyacrylonitrile fibers was treated with an aqueous bath which contained, per liter, about 40 parts of the emulsion described in Example 1 of a reaction product containing isocyanate groups and about 20 parts of the about 40% aqueous emulsion of an organopolysiloxane of the formula



and about 0.3 part of zinc acetate. The fabric of polyacrylonitrile fibers was squeezed out to a bath uptake of about 70%, dried at about 130° C. and then treated with steam at about 102° C. for up to about 2 minutes. The fabric treated according to the invention has a pleasant, smooth handle and improved response to creasing.

	Dry creasing angle		Wet creasing angle	
	Warp	Weft	Warp	Weft
35 Untreated fabric.....	122°	125°	126°	132°
40 Fabric treated according to the invention.....	159°	165°	142°	150°
Abrasion resistance (repenning)				
Untreated fabric, revolutions.....	320			
Fabric treated according to the invention, revolutions.....	400			

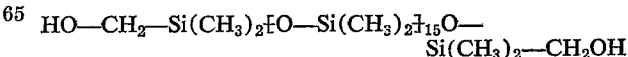
EXAMPLE 5

45 Wool yarns were treated with an aqueous bath which contained, per liter, about 60 parts of the emulsion of a reaction product containing isocyanate groups, which reaction product was prepared on the basis of an organopolysiloxane about 10 parts of the mixture described in Example 1 of the emulsion of an organopolysiloxane and the aqueous dispersion of a copolymer and about 0.2 part of zinc acetate.

50 The wool yarns were treated by immersion in the aqueous bath and centrifuged to reduce the bath uptake to about 50%. They were dried at about 115° C.

The wool yarn treated according to the invention has a pleasant, soft handle and has no tendency to felt after several mild washes. Shrinkage is considerably diminished. The emulsion of the reaction product which contains isocyanate groups and is based on an organopolysiloxane was prepared as follows:

About 1000 parts of an organopolysiloxane of the formula



containing about 3.7% by weight of hydroxyl groups were mixed with about 1000 parts of 1,6-hexamethylene diisocyanate and heated at about 120° C. for about 2 hours.

70 Excess diisocyanate was then removed at about 160° C. in a thin layer evaporator at about 0.1 mm. Hg. A 50% emulsion was then prepared from about 500 parts of the resulting reaction product, which contained about 5.7% by weight of free isocyanate groups, with the use of about

100 parts of ethyl acetate and about 400 parts of water with the addition of a commercial surface-active paraffin sulphionate.

EXAMPLE 6

Yarns consisting of about 55% of polyester fibers and about 45% wool were treated with an aqueous bath which contained, per liter, about 50 parts of the emulsion described in Example 1 of a reaction product which contains isocyanate groups, about 20 parts of the about 40% emulsion described in Example 1 of an organopolysiloxane and about 0.3 part of zinc acetate.

The yarns were treated with the aqueous bath by immersion centrifuged to reduce the bath uptake to about 60% and dried at about 130° C. The yarns treated according to the invention have a pleasant, soft handle and do not tend to felt or shrink when washed.

It is to be understood that the foregoing working examples are given for the purpose of illustration and that any other suitable active hydrogen containing compound organic polyisocyanate, vinyl compound or the like could be used in this process provided that the teachings of this disclosure are followed.

Although the invention has been described in considerable detail in the foregoing, it is to be understood that such detail is solely for the purpose of illustration and that many variations can be made by those skilled in the art without departing from the spirit and scope of the invention except as set forth in the claims.

What is claimed is:

1. The process for finishing a textile material to impart an improved hand and a reduced susceptibility to soiling which comprises treating a textile material with an aqueous bath which consists of essentially of (1) an isocyanato prepolymer prepared by a process which comprises reacting an organic polyisocyanate with an organic compound having a molecular weight of about 500 to about 10,000 and containing at least two active hydrogen containing groups as determined by the Zerewitinoff method (2) and 1 to 20 equivalent percent, based on said prepolymer of an organopolysiloxane having a molecular weight of up to about 25,000 and containing at least two active hydrogen containing groups as determined by the Zerewitinoff method; and reacting said prepolymer (1) and said polysiloxane (2) in situ on said textile material.

2. The process of claim 1 wherein said aqueous bath additionally contains 0.1 to 10 g. per liter of water-soluble lower carboxylic acid salts of metals of Groups I, II or VIII, of the Periodic System of elements, and wherein after treating with said aqueous bath; said textile material is dried and then treated for 3 to 30 minutes in water at 10°-100° C. or for ½-10 minutes with steam at 100°-110° C., said water optionally including 1-5 g. of hydro-

gen peroxide or 0.1-1 g. of catalysts which accelerate the reaction of isocyanate groups with compounds containing active hydrogen atoms.

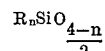
3. A textile material treated according to the process of claim 1.

4. A textile material treated according to the process of claim 1 wherein said aqueous composition also contains 0.1-10 g. per liter of a water-soluble lower carboxylic acid salt of a metal of Groups I, II, or VIII of the Periodic System of elements.

5. The process of claim 1 wherein polymers or copolymers prepared from vinyl or divinyl monomers are included additionally in the said aqueous bath.

6. A textile material treated according to the process of claim 5.

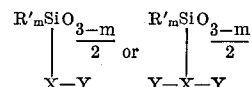
7. The process of claim 1 wherein said organopolysiloxane has the general formula



in which n is greater than 1 and not more than 3 and in which each substituent R is one of the radicals R' , $-X-Y$, or



R' representing an aliphatic, cycloaliphatic or aromatic hydrocarbon radical, X is an aliphatic or araliphatic bivalent or trivalent organic radical which may contain ether, thioether, ester, or amino group and Y is a hydroxyl, sulphhydryl, carboxyl, carbonamide, or secondary amino group; at least two siloxane units of said polysiloxane being of the formula



in which R' , Y and X have the meaning indicated above and m is the integer 1 or 2, and the substituents R in the remaining siloxane units being exclusively the radicals R' .

8. A textile material treated according to the process of claim 7.

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