

1

2

3,529,990

**PROCESS OF FINISHING TEXTILE MATERIALS**  
 Gustav Becker, Leverkusen, Heinz Griepentrog, Cologne,  
 Wolfgang Klebert and Friedrich Reich, Leverkusen,  
 and Karl Schafer, Opladen, Germany, assignors to  
 Farbenfabriken Bayer Aktiengesellschaft, Leverkusen,  
 Germany, a corporation of Germany  
 No Drawing. Filed Sept. 22, 1967, Ser. No. 669,711  
 Claims priority, application Germany, Sept. 24, 1966,  
 F 50,279

Int. Cl. B44d 1/44

U.S. Cl. 117—63

6 Claims

## ABSTRACT OF THE DISCLOSURE

Textile materials are treated with polymers based on vinyl monomers and polyurethane prepolymers having free —NCO groups in the presence of metal salts of carboxylic acids, drying the treated textile material if desired and then treating it with water.

This invention is concerned with a process for the production of finishes of textile materials and in particular with the production of urethane finishes of textile materials which have improved properties.

It has been proposed heretofore to coat textiles with both polyester and polyether urethanes for various purposes including the shrink proofing of wool and the like. However, the heretofore known coatings and finishes have not been entirely satisfactory because the heretofore known textile finishing process often leads to unsatisfactory tear resistance in the finished textile material. Furthermore, the heretofore known textiles are often unsatisfactory with respect to crease resistance, abrasion resistance and retention of shape. The heretofore known coated textiles based on mixtures of vinyl polymers and polyurethanes are sticky and change on storage. Also they have poor bond.

It is therefore an object of this invention to provide improved finished textile material. Another object of this invention is to provide textile materials and finishes therefor which have improved tear strength. A further object of this invention is to provide improved textile materials which have improved crease resistance, abrasion resistance and improved ability to retain their shape. 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, in addition to polymers or copolymers prepared from vinyl or divinyl monomers and in addition to reaction products containing isocyanate groups, which reaction products are prepared from compounds of molecular weight of 500 to 10,000 which carry at least two active hydrogen atoms and/or organopolysiloxanes which carry at least two active hydrogen atoms and have molecular weights of up to 25,000 and polyisocyanates, an addition of 0.1 to 10 g. per liter of water-soluble salts of metals of sub-Group I, II or VIII of the Periodic System of elements with lower mono- or polycarboxylic acids, drying the textile materials if desired and then subjecting them to an after-treatment with water.

This after-treatment may, for example, be either ½ to 10 minutes treatment with steam at 100° C. to a maximum of 110° C. or 3 to 30 minutes treatment with water at 10 to 100° C.; if the treatment with water is

carried out at temperatures below 60° C., it is preferred to add to the water, per liter, 1 to 5 g. of 30% aqueous hydrogen peroxide or 0.1 to 1 g. of catalysts which accelerate the isocyanate reaction with compounds containing active hydrogen atoms. 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 acetonates of nickel, cobalt or iron. It is also found in many cases to be advantageous to add surface active agents in an amount of 0.5 to 5 g. per liter.

The after-treatment in water may be carried out by simple immersion in which the textile materials are thoroughly wetted or as a washing process. The after-treatment takes between 3 and 30 minutes, generally 15 to 20 minutes.

In practice, the methods employed for the after-treatment according to the invention and the variations thereof employed will be selected from case to case according to the given conditions.

After-treatment of the impregnated and dried textile materials may directly follow drying, but may equally well be carried out 24 hours later, and there is therefore ample scope for variation of the time factor so that best use can be made of the plant available to achieve optimum production.

The salts chosen from sub-Group I, II or VIII of the Periodic System of elements are e.g., Zn, Cd, Hg, Cu, Ag, Au, Fe, Co, Ni, Ru, Rh, Pd, Os, Ir and Pt and are preferably zinc, cadmium, copper or nickel. Acids suitable for salt formation are, for example, lower carboxylic acids such as formic acid, acetic acid, trichloroacetic acid, propionic acid, butyric acid, valeric acid, lactic acid, malonic acid and glutaric acid. Preferred additives are the zinc salts of lower monocarboxylic acids, such as zinc acetate or zinc propionate. The salts are used in quantities of 0.1 to 10 g., preferably 0.2 to 1 g. per liter of bath.

The following are examples of vinyl or divinyl monomers on which the polymers or copolymers to be used according to the invention may be based: ethylene, propylene, vinyl chloride, vinyl acetate, vinyl ether, styrene or divinyl benzene, butadiene, isoprene or chloroprene and  $\alpha,\beta$ -unsaturated carboxylic acids such as acrylic acid or methacrylic acid as well as their nitriles, esters and amides. Polymers which have groups capable of reacting with isocyanates are generally to be preferred; these include, for example, the polymers or copolymers prepared from acrylic acid, methacrylic acid and their hydroxy-alkyl esters or amides, as well as the copolymers which are obtained when N-methylol-acrylamide or 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, for example by the process according to U.K. patent specification 1,002,451.

Examples of polymeric organic compounds of molecular weight 500 to 10,000 which contain active hydrogen atoms, i.e. active hydrogen atoms as determined by the Zerewitinoff method, and which form the basis of the reaction products according to the invention which contain isocyanate groups are polyalkylene glycols such as polyethylene, polypropylene, polybutylene and polyhexylene glycols or copolymers and graft polymers of the alkylene oxides from which these polyethers are derived, or polythioethers which are obtainable by condensation of thiodiglycol with itself or with sulphur-free polyalcohols. Polyesters obtained by condensation of dicarboxylic acids with polyalcohols or by condensation of hydroxycarboxylic acids can also be used. In addition, polyester

3

amides such as described in U.K. patent specification 580,524 and polyacetals such as described in U.K. patent specification 840,097 may be used.

All these higher molecular weight organic compounds preferably have two or more free hydroxyl groups, but compounds with free carboxyl, mercapto or amino groups are also suitable. These compounds may have molecular weights of up to 10,000 although the preferred range lies between 750 and 5000.

In addition to the high molecular weight compounds containing active hydrogen atoms, smaller quantities of lower molecular weight compounds, e.g. diols, which have a modifying effect on the properties of the end products, may also be used.

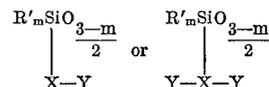
Other compounds suitable for the reaction products which contain isocyanate groups are organopolysiloxanes containing at least two active hydrogen atoms, which polysiloxanes have the following general formula:



in which  $n$  is greater than 1 and at the most 3 and in which each substituent  $R$  is one of the radicals  $R'$ ,  $-X-Y$  or  $Y-X-Y$ , in which  $R'$  represents an aliphatic, cycloaliphatic or aromatic hydrocarbon radical, which may carry an inert substituent, and  $X$  denotes an aliphatic or araliphatic bivalent or trivalent radical which may contain an ether, thioether, ester or amino group, and  $Y$  stands for a hydroxyl, sulphhydryl, carboxyl or carbonamide group or for a secondary amino group,

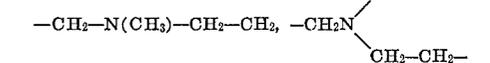
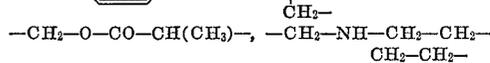
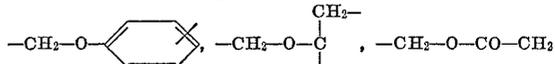
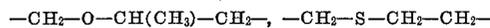
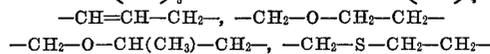
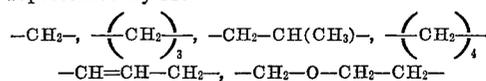
4

with the proviso that at least two siloxane units correspond to the formula

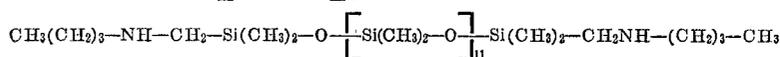
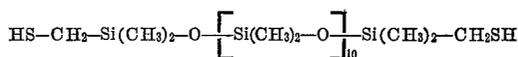
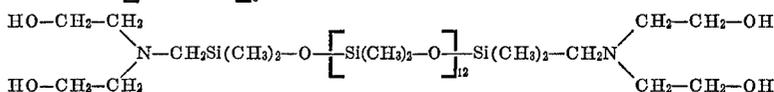
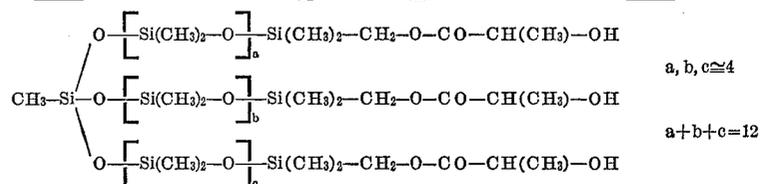
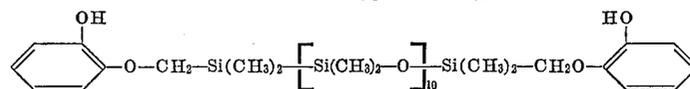
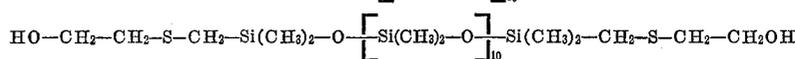
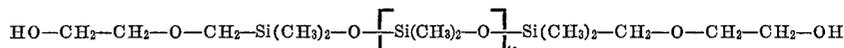
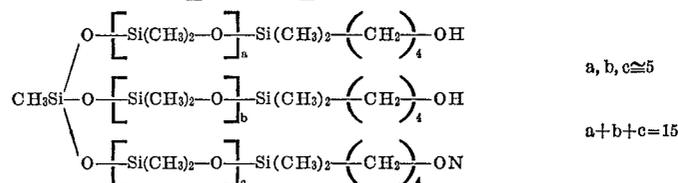
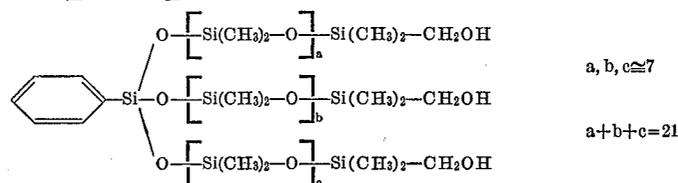
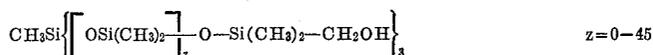
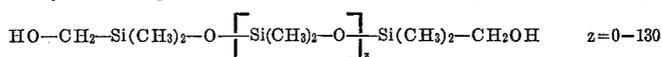


in which  $R'$ ,  $X$  and  $Y$  have the meaning indicated and  $m$  is the integer 1 or 2, and the substituents  $R$  in the remaining siloxane units are exclusively the radicals  $R'$ .

The following are examples of hydrocarbon radicals represented by  $R'$ : methyl, ethyl, isopropyl, tertiary butyl, octadecyl, allyl, chloromethyl, trifluoropropyl, cyclohexyl, phenyl and tolyl; preferred are polysiloxanes of the given formula wherein  $R'$  stands for methyl or phenyl. The following are examples of bivalent or trivalent radicals represented by  $X$ :



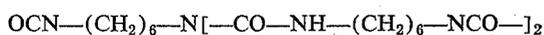
The following are given as representative examples of polysiloxanes of the general formula given:



Polysiloxanes of this type are already known; they have been described, e.g. in German patent specification 1,199,772, in German published specification 1,196,869, in British patent specifications 980,778 and 995,394 and in French patent specifications 1,361,115 and 1,365,050.

Small quantities of monofunctional organopolysiloxanes may also be used in addition to the above compounds.

The polyisocyanates used for the preparation of the reaction products are preferably aliphatic and cycloaliphatic diisocyanates. Examples are tetramethylene diisocyanate, hexamethylene diisocyanate, 1,4-cyclohexane diisocyanate, 4,4'-dicyclohexylmethane diisocyanate and 2,4- and 2,6-hexahydrotoluylene diisocyanate. Aromatic diisocyanates are also suitable, e.g. p-phenylene diisocyanate and 2,4- or 2,6-toluylene diisocyanate, as well as triisocyanates such as the reaction product of the formula



obtainable from 3 mols. of hexamethylene diisocyanate and 1 mol. of water. The reaction products which contain isocyanate groups may be prepared by the known methods of reacting the compounds carrying at least two active hydrogen atoms with a stoichiometric excess of polyisocyanates at elevated temperatures. The reaction products containing isocyanate groups may also be reacted in the form of the known bisulphite addition products.

To prepare the impregnating baths, inert, non-hydrophilic solvents such as benzene, toluene, ethylacetate, benzene or chlorinated hydrocarbons may be added to the reaction products containing isocyanate groups of the type mentioned above in order to reduce their viscosity to a low value, and the reaction products may then be emulsified, with the use of a high speed stirrer, in water which contains 0.5 to 10% of suitable emulsifiers in addition to the salts used according to the invention. The polymers or copolymers to be used are then stirred into this emulsion, usually in the form of their aqueous dispersions, and the bath is then diluted to the desired concentration with water.

crease has been reduced to about 40 to 150%, preferably 70 to 100%, shaking out excess liquid or removing it by suction, drying and, as explained above, after-treating. Alternatively, the textile materials may be sprayed with the aqueous baths and then dried and after-treated.

Excellent finishes can be obtained by the process according to the invention on textile materials of natural or synthetic origin, for example textile materials of cotton, regenerated cellulose, wool and silk, polyamide, polyurethane, polyester, polycarbonate, polyacrylonitrile or polypropylene fibers, especially as regards crease-resistance, abrasion-resistance and dimensional stability. A special advantage of the process according to the invention is that after the impregnation, optional drying, and after-treatment, the textile materials are dry and not sticky. They do not undergo any further change on storage. The hand of the finish can be varied to the desired extent from soft to hard by choice and composition of the two components, i.e. the polymer or copolymer emulsions and the reaction products which contain isocyanate groups. The finished textiles are useful to make clothing and the like.

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

#### EXAMPLE 1

A mixed fabric of about 55% polyacrylonitrile fibers and about 45% wool is treated with an aqueous bath which contains, per liter, about 20 g. of a 40% aqueous dispersion of a copolymer of about 80 parts of butyl acrylate, about 10 parts of styrene and about 10 parts of acrylamide as well as about 30 g. of the emulsion described in the last paragraph of this example and 0.3 g. of zinc acetate.

The mixed fabric of polyacrylonitrile fibers and wool is squeezed out to a bath uptake of about 90%, dried for about 10 minutes at about 100 to 110° C., and then treated for up to about one minute with steam at about 102° C. The fabric treated according to the invention has a full, pleasant hand and the following advantages compared to an untreated fabric.

	Dry creasing angle		Wet creasing angle		Percent shrinkage machine after 5 washes, 30° C.		Appearance of fabric	Abrasion resistance revolutions
	K, °	S, °	K, °	S, °	K	S		
Untreated fabric.....	130	132	121	120	-4.2	-3.8	Felted.....	430
Fabric treated according to the invention.....	162	168	140	145	-0.6	-0.5	Not felted, clear and open....	580

Emulsifiers suitable for the preparation of the impregnating baths are those of the ionic or non-ionic types which yield oil-in-water emulsions without exerting a too powerful catalytic effect on the isocyanate-water reaction. Emulsifiers used include the sodium salts of alkyl sulphonates, alkyl aryl sulphonates and the acidic sulphuric acid esters of higher aliphatic alcohols as well as the hydroxy-ethylation products of long chained alkyl phenols.

The addition of organic solvents may be dispensed with under certain conditions, e.g. if the isocyanate prepolymers have a very low viscosity. The baths may also be prepared by other methods, e.g. via a water-in-oil emulsion which reverses on addition of excess water.

The proportion by weight of polymers or copolymers to reaction products containing isocyanate groups should preferably be 0.25-4:1. The amount of polymers or copolymers and reaction products containing isocyanate groups in the aqueous bath may also vary within wide limits, say between 2 and 70% by weight.

The textile materials may be treated with the aqueous baths used according to the invention by steeping them in the given aqueous baths, squeezing out until the weight in-

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

About 3000 g. of a branched polypropylene glycol which had molecular weight of about 3000, OH number about 55.5 and acid number about 0.5 was heated with about 535 g. hexamethylene diisocyanate for about 2 hours at about 110° C. and then for a further 1½ hours at about 130 to 140° C. From about 500 g. of the resulting reaction product which contained about 3.9% by weight of free isocyanate groups, an emulsion was then prepared with about 100 g. ethyl acetate and about 400 g. water with the addition of about 4 g. of a paraffin sulphonate.

#### EXAMPLE 2

Packages of wool yarns were treated with aqueous dispersions the copolymers described in Example 1 and with the emulsion described in Example 1 of a reaction product containing isocyanate groups as well as with the emulsion described below of a reaction product containing isocyanate groups but based on an organopolysiloxane. The bath contained in addition about 0.3 g. per liter of copper acetate.

The quantities of emulsions used were as follows, based on the weight of wool yarns:

10% of the emulsion of the reaction product described in Example 1 which contains isocyanate groups

5% of the emulsion described below of a reaction product containing isocyanate groups, which reaction product is based on an organopolysiloxane

15% of the aqueous dispersion of a copolymer described in Example 1.

The treatment was carried out by pumping the bath liquor through the packages alternately from inside outwards and from outside inwards.

The packages were treated for about 40 minutes at about 20° C. A mixture of glycollic acid and stannous chloride in the ratio of about 1:1.5 was then added in an amount of about 0.2 g. per liter of treatment baths and treatment was continued for a further 20 minutes at about 20° C.

Excess liquid was then extracted from the packages of wool yarn and the yarn was dried at about 100° C. The wool yarns treated as described above have the following properties:

Shrinkage after 3 machine washes at 60° C.:

Untreated yarn—22%

Yarn treated according to the invention—0.6% felting

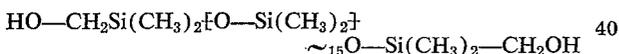
Untreated yarn—Completely felted

Yarn treated according to the invention—Not felted, open

The yarn treated according to the invention has a pleasant, woolly hand and is eminently suitable for further working up into woven fabrics or knitted goods.

The emulsion of reaction product based on an organopolysiloxane and containing isocyanate groups was prepared as follows:

About 1000 g. of an organopolysiloxane of the formula



containing about 2.7% by weight of hydroxyl groups were mixed with about 1000 g. of 1,6-hexamethylene diisocyanate and heated for about 2 hours at about 120° C. Excess diisocyanate was then distilled off at about 160° C. in a thin layer evaporator at a pressure of about 0.1 mm. Hg. From about 500 g. of the resulting reaction product which contained about 5.7% by weight of free isocyanate groups, a 50% emulsion was then prepared as described in Example 1 with about 100 g. ethyl acetate and about 400 g. water with the addition of about 4 g. of a surface active paraffin sulphonate.

### EXAMPLE 3

Packages of wool yarns were treated as described in Example 2 with aqueous dispersions of copolymers and reaction products containing isocyanate groups.

Instead of about 0.2 g. of the mixture of glycollic acid and stannous chloride of Example 2, there were added at the rate of about 4 g. per liter an emulsion which was prepared as follows:

About 5 g. of dibutyl tin laurate were emulsified with about 15 g. of ethyl acetate, about 88 g. of water and about 1 g. of paraffin sulphonate by means of a high speed stirrer. The packages were treated with this emulsion for about 20 minutes at about 20° C. and surplus

liquid was centrifuged off or removed by suction and the packages were dried at about 100° C.

The wool yarns treated as described above underwent about 0.7% shrinkage after three machine washes at about 60° C. and have a pleasant soft hand and are not felted.

### EXAMPLE 4

Combed wool sliver was padded with an aqueous dispersion which contained per liter about 20 g. of a 40% aqueous dispersion of a copolymer of about 60 parts butyl acrylate, about 30 parts styrene and about 10 parts acrylamide, and about 60 g. of the emulsion described in Example 1 of a reaction product containing isocyanate groups, and about 0.4 g. zinc propionate. The sliver was squeezed out to reduce the liquor uptake to about 80%, and dried at about 110° C. It was then treated for several minutes with steam at about 101 to 102° C.

The yarns made from the combed wool sliver treated according to the invention show no felting after several washes at about 60° C. and the shrinkage is much less than in the untreated material.

### EXAMPLE 5

A fabric of polyacrylonitrile fibers is treated with an aqueous dispersion which contains per liter about 10 g. of the dispersion of a copolymer described in Example 4 and about 30 g. of the emulsion of a reaction product containing isocyanate groups described below and about 0.5 g. zinc acetate. The fabric is squeezed out to a liquor uptake of about 60%, dried at about 120° C. for about 10 minutes and then treated for several minutes with steam at about 102° C.

The fabric treated according to the invention has a full, dry hand and has undergone excellent improvement in its crease-resistance, as shown in the following comparison:

	Dry creasing angle		Wet creasing angle	
	K,°	S,°	K,°	S,°
Untreated fabric.....	120	121	125	130
Fabric treated according to the invention	160	162	145	155

The reaction product based on a thioether and containing isocyanate groups was prepared as follows:

About 500 g. of a polythioether prepared by condensation of about 700 parts of thiodiglycol and about 300 parts of 2,2-dimethylpropanediol-(1,3) to an average molecular weight of about 1900 (OH number 59) were heated with about 500 g. of hexamethylene diisocyanate for about one hour at about 110° C. The excess diisocyanate was then removed from the reaction mixture by distillation at temperatures of up to about 150° C. at a pressure of about 0.1 mm. Hg. From about 500 g. of the resulting reaction product which had a free isocyanate group content of about 4.3% by weight, a 50% emulsion was then prepared with about 100 g. ethyl acetate and about 400 g. water with the addition of about 4 g. of a surface active paraffin sulphonate.

### EXAMPLE 6

Fabrics of polyester fibers and polyamide fibers were treated according to Example 5.

The fabrics treated according to the invention have a full, non-sticky hand and the following advantages in their response to creasing:

	Fabrics of polyester fibers				Fabrics of polyamide fibers			
	Dry creasing angle		Wet creasing angle		Dry creasing angle		Wet creasing angle	
	K,°	S,°	K,°	S,°	K,°	S,°	K,°	S,°
Untreated fabric.....	130	141	140	141	111	115	95	110
Fabric treated according to the invention.....	162	165	155	158	165	167	160	162

9

## EXAMPLE 7

A cotton fabric is treated with an aqueous bath containing about 100 g. of the dispersion of a copolymer described in Example 4, about 100 g. of the emulsion described in Example 1 of a reaction product containing isocyanate groups and about 90 g. of dimethyloldihydroxyethylene urea, about 10 g. magnesium chloride and about 0.5 g. zinc acetate. The fabric is squeezed out to reduce the liquor uptake to about 90% and is heated at about 150° C. for about 5 minutes. It is then treated for several minutes with steam at about 102° C.

The fabric treated according to the invention has a pleasant, full hand and, as will be seen from the following comparison, excellent crease resistance and improved abrasion resistance.

	Dry creasing angle		Wet creasing angle		Abrasion resistance, revolutions
	K, °	S, °	K, °	S, °	
Untreated fabric.....	90	91	82	80	250
Fabric treated according to the invention.....	145	150	145	155	380

## EXAMPLE 8

Prices of knitted wool were treated with an aqueous bath containing per liter about 20 g. of the dispersion of a copolymer described in Example 4 and about 60 g. of the emulsion of the reaction product containing isocyanate groups described in Example 1 and about 0.3 g. zinc acetate. The knitted pieces were centrifuged to reduce the liquor uptake to about 70%, dried at about 110° C. and then treated at about 40° C. with a bath containing about 3 g. of a 30% hydrogen peroxide solution per liter. After a treatment time of about 15 minutes, the pieces were dyed with acid dyes in the same bath.

In contrast to untreated pieces of knitted wool, the knitted goods treated according to the invention do not felt in the dyeing process, nor do they show any signs of

Treatment immediately after preparation of the baths	Percent shrinkage of the wool fabric				Felting of wool fabric				Stability of baths	
	Treatment 4 hours after preparation of the baths		Treatment immediately after preparation of the baths		Treatment 4 hours after preparation of the baths		Stability of baths			
	Warp	Weft	Warp	Weft	Not felted.....	Not felted.....	Immediately after preparation	4 hours after preparation		
(a).....	0.4	0.3	0.5	0.4	(a) Not felted.....	Not felted.....	(a) No change.....	No change.		
(b).....	1.1	1.2	3.9	3.8	(b) Not felted.....	Strongly felted.....	(b) No change.....	Formation of skin and ground deposit		
(c).....	1.3	1.4	4.6	5.8	(c) Slightly felted.....	do.....	(c) No change.....	Do.		

felting after 10 mild washes in a drum washing machine, and they shrink much less than the untreated goods.

## EXAMPLE 9

A wool georgette is treated with an aqueous bath containing per liter about 40 g. of the aqueous dispersion of a copolymer described in Example 4 and about 80 g. of the emulsion, described in Example 1, of a reaction product containing isocyanate groups, and about 0.4 g. zinc acetate. The fabric is squeezed out to reduce the liquor uptake to about 80% and is dried at about 110° C. The wool georgette is treated in a winch vat with about 2 g. per liter of hydrogen peroxide (30%) at about 40 to 50° C. for about 15 minutes and is then dyed in the same bath.

In contrast to an untreated fabric, the wool georgette treated according to the invention does not show any tendency to felt even after prolonged dyeing and it has no tendency to form swellings during the dyeing process. Compared with an untreated wool material, the article treated according to the invention shows a remarkable saving of dye in the dyeing process.

After 3 machine washes at about 60° C., the wool georgette treated according to the invention has undergone

10

a shrinkage of about 0.8% in the direction of the warp and about 0.6% in the direction of the weft and is not felted. An untreated material shrinks after three machine washes at about 60° C. by about 16% in the direction of the warp and about 8% in the direction of the weft and is felted.

## EXAMPLE 10

A wool cloque was treated with the baths from Example 9 and then dried at about 110° C. and treated for about 2 minutes with steam at about 102 to 103° C.

The fabric treated in this way is not felted and has undergone much less shrinkage than an untreated fabric.

## EXAMPLE 11

A wool fabric is treated with an aqueous bath containing per liter about 40 g. of a 40% aqueous dispersion of a copolymer of about 60 parts of butyl acrylate, about 30 parts of styrene and about 10 parts of acrylamide as well as about 80 g. of the aqueous dispersion described in Example 1 of a reaction product containing isocyanate groups.

In addition, about

- (a) 0.3 g. zinc acetate or
- (b) 0.3 g. tin octoate or
- (c) 0.3 g. triethylenediamine

were added per liter to the aqueous bath.

The tin octoate was added in the form of a 5% solution in ethyl acetate, and the solution was emulsified in the bath with the aid of a commercial paraffin sulphonate.

Cuttings of wool fabric were treated with the aqueous baths immediately and after the bath compositions had been left to stand for about 4 hours.

The wool fabric was squeezed out to reduce the liquor uptake to about 90% and was dried for about 15 minutes at about 110 C. It was then treated with steam at about 102° C. for about 2 minutes. The cuttings of wool fabric treated in this way were then subjected to a machine wash at about 60° C.

The hand of the treated wool cuttings is in all cases pleasant and not sticky. Only zinc acetate, however, is found to be suitable as catalyst in the sense that it keeps the aqueous baths usable for prolonged periods and is necessary for the finishing of textiles.

It is to be understood that the foregoing examples are given for the purpose of illustration and that any other suitable surface active agent, active hydrogen containing group or vinyl copolymer can be used therein provided that the teachings of this disclosure are followed.

What is claimed is:

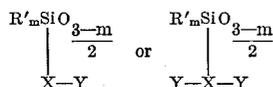
1. The process of finishing textile materials which comprises applying to said textile materials an aqueous liquor containing (1) a polymer or copolymer based upon a monomer selected from the group consisting of ethylene, propylene, vinyl chloride, vinyl acetate, vinyl ether, styrene, divinyl benzene, butadiene, isoprene, chloroprene,  $\alpha$ ,  $\beta$  unsaturated carboxylic acids,  $\alpha$ ,  $\beta$  unsaturated carboxylic acid nitriles,  $\alpha$ ,  $\beta$  unsaturated carboxylic acid esters, and  $\alpha$ ,  $\beta$  unsaturated carboxylic acid amides, (2) a reaction product containing free isocyanate groups prepared from (a) an organic compound carrying at least two active hydrogen atoms selected from the group consisting of a compound of molecular weight between

500 and 10,000, an organo polysiloxane of molecular weight up to 25,000 and mixtures thereof with (b) a polyisocyanate, and (3) 0.1 to 10 grams per liter of a water soluble salt selected from the group consisting of water soluble salts of metals selected from the group consisting of zinc, cadmium, mercury, copper, silver, gold, iron, cobalt, nickel, ruthenium, palladium, osmium, iridium, platinum and rhodium with low molecular weight mono or polycarboxylic acid and after treating the textile material with steam at 100 to 110° C. or water at between 10 and 100° C., the proportion of polymer or copolymer (1) to product (2) being about 0.25 to 4:1 by weight and the amount of (1) and (2) in the aqueous liquor being between 2 and 70% by weight.

2. The process of claim 1 wherein the reaction product having free isocyanate groups is prepared from organopolysiloxane of the general formula



wherein  $n$  is greater than 1 and not greater than 3 and  $R$  is chosen so that at least two of the siloxane units correspond to the formulae



and in all of the remaining siloxane units  $R=R'$ , wherein  $m$  is 1 or 2 and  $R'$  is selected from the group consisting of saturated aliphatic hydrocarbon radicals, unsaturated aliphatic hydrocarbon radicals, cycloaliphatic hydrocarbon radicals, aromatic hydrocarbon radicals, substituted saturated aliphatic hydrocarbon radicals, substituted unsaturated aliphatic hydrocarbon radicals, substituted cycloaliphatic hydrocarbon radicals, and substituted aromatic hydrocarbon radicals wherein said substituents are inert,  $X$  is selected from the group consisting of aliphatic divalent radicals, aliphatic trivalent radicals, araliphatic divalent radicals, araliphatic trivalent radicals, substituted aliphatic divalent radicals, substituted aliphatic trivalent

radicals, substituted araliphatic divalent radicals, and substituted araliphatic trivalent radicals wherein the substituent are selected from the group consisting of ether, thioether, ester and amino groups, and  $Y$  is selected from the group consisting of hydroxyl, sulphhydryl, carboxyl, carbonamide or secondary amino groups.

3. The process of claim 1 wherein said aqueous bath contains from about 0.5 to about 5 grams per liter of a surface active agent.

4. The process of claim 1 wherein the after-treatment is accomplished with steam.

5. The process of claim 1 wherein the after-treatment is accomplished with water with the addition of a catalyst to accelerate the reaction of the isocyanate groups with compounds containing active hydrogen atoms.

6. The process of claim 1 wherein the organic polyisocyanate is an aliphatic or cycloaliphatic diisocyanate.

#### References Cited

##### UNITED STATES PATENTS

2,753,276	7/1956	Brochhagen	-----	117-161	X
2,901,467	8/1959	Croco	-----	117-161	X
3,135,711	6/1964	Thoma	-----	117-161	X
3,301,644	1/1967	Habib	-----	117-161	X
2,917,412	12/1959	Reinhardt et al.	----	117-139.4	
2,929,737	5/1960	Tischbein	-----	117-76	
3,238,010	3/1966	Habib et al.	-----	8-115.6	
3,318,659	5/1967	Bullock et al.	-----	8-115.6	

##### FOREIGN PATENTS

745,960	3/1956	Great Britain.
565,889	11/1958	Canada.

WILLIAM D. MARTIN, Primary Examiner

W. R. TRENOR, Assistant Examiner

U.S. Cl. X.R.

8-115.6; 117-139.5, 145; 252-8.6; 260-46.5, 77.5