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**POLYURETHANE-FORMING COMPOSITIONS AND PROCESS FOR PREPARING POLYURETHANE-BACKED TEXTILES**

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(57) Claim

1. A process for preparing a polyurethane-backed textile, comprising the steps of

(A) applying to a surface of a textile a layer of a polyurethane-forming composition comprising

(1) a polyol based on a $C_3$-$C_8$ alkylene oxide, which polyol has an equivalent weight of from 1800 to 3000, and an internal poly(ethylene oxide) block or a terminal ethylene oxide cap constituting from 15 to 30 percent of the weight of the polyol, or mixture of such polyols, wherein said polyol or mixture thereof has an average functionality of from 1.8 to 2.2,

(2) from 5 to 25 parts, per 100 parts by weight of component (1), of a low equivalent weight compound having 2 active hydrogen-containing groups per

.../2

molecule, and

(3) a polyisocyanate having an average functionality of from 1.9 to 2.4, in an amount to provide the composition with 0.9 to 1.25 isocyanate groups per active hydrogen-containing group, and

(4) an effective amount of a blowing agent and

(B) curing said polyurethane-forming composition to form a cellular polyurethane backing adherent to said textile.

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Complete Specification for the invention entitled:

POLYURETHANE-FORMING COMPOSITIONS AND PROCESS FOR PREPARING POLYURETHANE-BACKED TEXTILES

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The following statement is a full description of this invention, including the best method of performing it known to applicant(s):

# POLYURETAHNE-FORMING COMPOSITIONS AND PROCESS FOR PREPARING POLYURETHANE-BACKED TEXTILES

This invention relates to polyurethane-backed textiles, and particularly to textiles which are backed with a polyurethane foam.

Various textiles, most notably carpet, often have an attached polyurethane foam backing for cushioning and other purposes. These polyurethane-backed textiles are usually prepared by applying to one surface of the textile a mixture of a polyisocyanate and at least one high equivalent weight polyol, which subsequently reacts to form a cellular backing adherent to the textile.

Preparing the backed textiles in this manner has met with formidable processing difficulties. As all of the reactive components of the polyurethane-forming composition are mixed prior to application to the textile, the initial reactivity must be delayed so that the composition does not gel before it is applied and gauged. The characteristics of the composition must also be such that it can easily be blown or

frothed to a desirable density. Once the composition is blown or frothed and applied to the carpet, it must react quickly and completely in order to form a tack-free backing in as short a cure time as possible. In addition, the resulting polyurethane backing must have good physical properties, particularly good tensile, elongation and tear properties, low residual tack and high dimensional stability. Often, modifications in a polyurethane-forming composition which improve one of these processing characteristics of physical properties make others worse. Thus, it has been very difficult to provide a polyurethane-forming composition which has all of the desired attributes in processing and physical properties.

Accordingly, it would be desirable to provide a process for preparing polyurethane-backed textiles, wherein a polyurethane-forming composition is employed which has improved processing characteristics and/or physical properties.

In one aspect, this invention is an improvement in a process for preparing a polyurethane-backed textile wherein an uncured polyurethane-forming composition is applied to a surface of the textile, gauged and cured to form an adherent backing thereto, the improvement comprising employing a polyurethane-forming composition comprising

(A) a polyol based on a $C_3$-$C_8$ alkylene oxide, which polyol has an equivalent weight of 1800 to 3000, and an internal poly(ethylene oxide) block or terminal ethylene oxide cap constituting from 15 to 30 percent of the weight of the polyol, or mixture of such



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polyols, wherein said polyol or mixture thereof has an average functionality from of 1.8 to 2.2,

(B) from 5 to 25 parts per 100 parts by weight of component (1) of a low equivalent weight

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polyols, wherein said polyol or mixture thereof has an average functionality from of 1.8 to 2.2,

(B) a minor amount of a low equivalent weight compound having 2 active hydrogen-containing groups per molecule, and

(C) a polyisocyanate having an average functionality of from 1.9 to 2.4, in an amount to provide the composition with from 0.9 to 1.3 isocyanate groups per active hydrogen-containing group, and

(D) an effective amount of a blowing agent.

In another aspect, this invention is a process for preparing a polyurethane-backed textile, comprising the steps of

(A) applying to a surface of a textile a layer of a polyurethane-forming composition according to this invention, and

(B) curing said polyurethane-forming composition to form a cellular polyurethane backing adherent to said textile.

In still another aspect, this invention is an active hydrogen-containing composition comprising

(A) a polyol based on a $C_3$-$C_8$ alkylene oxide, which polyol has an equivalent weight of from 1800 to 3000 and an internal poly(ethylene oxide block) or a terminal ethylene oxide cap constituting from 15 to 30 percent of the weight of the polyol, or mixture of such

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polyols, wherein said polyol or mixture thereof has an average functionality of from 1.8 to 2.2,

(B) from 5 to 25 parts, per 100 parts by weight of component (1) of a low equivalent weight

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polyols, wherein said polyol or mixture thereof has an average functionality of ~~from 1.8~~ to 2.2,

~~(B)~~ a ~~minor~~ amount of a low equivalent weight ~~compound~~ having 2 active hydrogen-containing groups per molecule, and

(C) from 5 to 500 parts, per 100 parts of component (A), of an inorganic, particulate filler.

In preparing polyurethane-backed carpet according to this invention, excellent processability and excellent physical properties of the resultant backed carpet are achieved.

In this invention, a polyurethane-forming composition comprising a certain high equivalent weight polyol, a chain extender and a polyisocyanate is used to provide a cellular textile backing.

The polyurethane-forming composition contains, as one component, a polyol based on a $C_3$-$C_8$ alkylene oxide. By "based on a $C_3$-$C_8$ alkylene oxide", it is meant that the polyol, exclusive of end-capping, is a polymer of a monomer or monomer mixture which is predominantly a $C_3$-$C_8$ alkylene oxide, although a minor amount of the monomer mixture, preferably not to exceed 30, preferably not to exceed 20 weight percent thereof, may be another copolymerizable monomer, preferably ethylene oxide. This polyol has an equivalent weight of from ~~1000 to 5000, preferably from~~ 1800 to 3000. ~~more preferably from 1900 to 2500.~~ The use of higher equivalent weight materials has been found to provide



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improved properties, notably improved tensile, elongation and tear properties.

The polyol also has an internal poly(ethylene oxide) block or a terminal ethylene oxide cap constituting from 15 to 30, preferably 16.5 to 30, more preferably from 17 to 25, most preferably from 17 to 20 percent of the weight of the polyol. It has been found that when polyols having equivalent weights of 1800 or higher are used, they are difficult to foam, especially in a frothing process, unless they contain an internal poly(ethylene oxide) block or terminal ethylene oxide cap constituting 15 weight percent or more of the polyol. However, if the ethylene oxide cap exceeds 30 weight percent, the polyol tends to be too hydrophilic to be suitable.

In addition, the polyol or, if a mixture of more than one such polyols are used, the mixture of polyols has an average functionality of from 1.8 to 2.2, preferably from 1.9 to 2.1, more preferably from 1.95 to 2.05, most preferably 1.98 to 2.02.

Preferably, the polyol comprises a polymer of propylene oxide which is end-capped with 17 to 20 weight percent ethylene oxide. Copolymer polyols which use such polyethers as the base polyol are also suitable. More preferably, the polyol is a mixture of a nominally trifunctional ethylene oxide-capped poly(propylene oxide) and a nominally difunctional ethylene oxide-capped poly(propylene oxide), in proportions such that the average functionality is within the recited ranges. Commercially available materials of this type have actual functionalities which are lower than the nominal functionalities, due

to the formation of monofunctional species during their manufacture. Accordingly, mixtures containing from 30 to 70, preferably from 40 to 60, weight percent of the nominal triol and 70 to 30, preferably from 60 to 40, weight percent of the nominal diol are preferred.

The polyurethane-forming composition also contains a chain extender, i.e., a low equivalent weight compound having 2 active hydrogen-containing groups per molecule. The active hydrogen-containing groups may be hydroxyl, mercaptan or amine, although if amine chain extenders are used, it is preferred that it be a sterically hindered, encapsulated or blocked amine as other amine chain extenders tend to be too reactive to be easily processed to make textile backings. Preferred chain extenders include, for example, the α,ω-alkylene glycols and glycol ethers such as ethylene glycol, 1,4-butane diol, 1,6-hexamethylene glycol, dipropylene glycol, tripropylene glycol, diethylene glycol and triethylene glycol; cyclohexanedimethanol; and the diverse bisphenols. Suitable amine chain extenders include, for example, methylene bis(o-chloroaniline), NaCl-blocked methylene dianiline and diethyltoluenediamine.

In making textile backings, a minor amount of chain extender is advantageously used. ~~Preferably~~ From 5 to 25, ~~more~~ preferably from 5 to 20 parts of the chain extender are used per 100 parts by weight of the polyol.

A polyisocyanate is used in the polyurethane-forming composition. The polyisocyanate used herein has an average functionality of from 1.9 to 2.4. Lower functionalities tend to provide an undercured

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polyurethane, whereas higher functionalities tend to promote trimerization of the polyisocyanate, which leads to undesirable crosslinking. When no precoat layer is used, the polyisocyanate preferably has an average functionality of from 1.95 to 2.2, more preferably from 1.95 to 2.1, in order that the polyurethane-forming composition as a whole has an average functionality of as close to 2.00 as possible. When a precoat layer is used, the polyisocyanate can have a somewhat higher functionality, as the precoat layer causes the textile to be more resistant to distortion due to crosslinking in the foam layer. In such case, the polyisocyanate preferably has an average functionality of from 2.0 to 2.3, more preferably from 2.03 to 2.2, as the slightly higher functionality provides a lower tack polyurethane having better properties.

The polyisocyanate may be aromatic or aliphatic. Suitable aromatic polyisocyanates useful herein include, for example, phenyl diisocyanate, 2,4- and/or 2,6-toluene diisocyanate, ditoluene diisocyanate, naphthylene 1,4-diisocyanate, 2,4'- and/or 4,4'-diphenylmethanediisocyanate and polymethylene polyphenylenepolyisocyanates. Suitable aliphatic polyisocyanates include, for example, the hydrogenated derivatives of the foregoing aromatic polyisocyanates, 1,6 hexamethylene diisocyanate, isophorone diisocyanate, 1,4-cyclohexyl diisocyanate and 1,4-bis(isocyanatomethyl)cyclohexane or mixtures thereof.

In addition, polyisocyanates containing urethane, urea, biuret, carbodiimide, allophanate and similar groups are also useful. Of particular interest

are the so-called "liquid MDI" products, having an equivalent weight of from 130 to 150, and prepolymers prepared from a diisocyanate and a relatively low equivalent weight compounds having from 2 to 4 hydroxyl groups per molecule.

Preferred are toluene diisocyanate (TDI) and diphenylmethanediisocyanate (MDI) and its derivatives, as well as prepolymers thereof. The most preferred TDI-based isocyanate is a prepolymer made by reacting an excess of TDI with glycerine, an adduct of glycerine and ethylene oxide or propylene oxide, trimethylolpropane, or mixtures thereof with a low equivalent weight polyoxyalkylene glycol such as diethylene glycol, tetraethylene glycol or higher polyethylene glycol so the resulting prepolymer has an average functionality of from 2.03 to 2.2. The most preferred MDI-based polyisocyanates are prepolymers of pure MDI and mixtures of MDI with a polymeric MDI having an average functionality of from 2.03 to 2.2.

The polyisocyanate is advantageously used in an amount sufficient to provide the polyurethane-forming composition with from 0.9 to 1.3, preferably from 1.05 to 1.25, more preferably from 1.05 to 1.2 isocyanate groups per active hydrogen-containing group. At lower levels of polyisocyanate, inadequate curing is achieved. At higher levels of polyisocyanate, increase crosslinking is obtained, which adversely affects the properties of the backing. It is noted that the optimum amount of polyisocyanate varies somewhat with the prevailing weather conditions, particularly temperature and humidity, as well as with the moisture

content of the textile substrate, as discussed in U.S. Patent No. 4,696,849.

In order to produce a cellular backing, a blowing agent is used. In this invention, the term blowing agent refers to any material which provides a cellular structure to the polyurethane. Suitable blowing agents include gasses such as, for example, air, nitrogen, carbon dioxide, argon and helium; water; low-boiling halogenated compounds such as the diverse chlorofluoromethanes and ethanes; the so-called azo blowing agents such as azobis(formamide) and finely divided solids. In this invention, it is preferred to employ a gas as the blowing agent, which is incorporated into the polyurethane-forming composition by frothing. The use of water in conjunction with the gas is also preferred, as the resulting foam contains urea linkages which improve tensile, elongation and tear strength. In any case, a sufficient amount of the blowing agent is used to provide a polyurethane backing having a density of from 2 to 55 pounds per cubic foot or more. For chemically blown backings, the density is preferably from 3 to 30, more preferably from 3 to 21 pounds per cubic foot. For frothed backings, the density is preferably from 10 to 30, more preferably from 10 to 18 pounds per cubic foot.

In addition to the aforementioned critical components, other optional ingredients may also be used. In particular, a catalyst for the reaction of the active hydrogen-containing materials with the polyisocyanate is preferably present, as is a surfactant which stabilizes the composition against cell collapse until cure is completed. It is also preferred to use an inorganic filler, in order to

provide improved physical properties and reduce the raw material cost. This filler is preferably inert, i.e., does not react with the active hydrogen-containing components or the polyisocyanate.

Suitable catalysts include organometallic catalysts and/or tertiary amine compounds. Suitable organometallic compounds include, for example, compounds of tin, lead, iron, bismuth and mercury. When a TDI-based polyisocyanate is used, the organotin and tertiary amine catalysts are preferred. Preferred organotin catalysts include stannic carboxylates such as tin octoate and tin oleate, and stannous compounds such as dimethyltindilaurate, dibutyltindilaurate, diethyltinediacetate, di(2-ethylhexyl) tin oxide, dialkyltinbis(alkylmercaptoesters) such as, for example, di(n-butyl)tinbis(isooctylmercaptoacetate) and di(isooctyl)tin bis(isooctylmercaptoacetate). Preferred tertiary amine catalysts include trialkyl amines and heterocyclic amines, such as alkylmorpholines, 1,4-dimethylpiperazine, triethylene diamine, bis(N,N-dimethylaminoethyl)ether, and N,N,N',N'-tetramethyl-1,3-butanediamine. When a MDI-based polyisocyanate is used, a delayed action catalyst is preferred. Organoiron or organobismuth catalysts are useful delayed action catalysts, but any other catalyst which gives delayed action is useful. Suitable organoiron catalysts include the iron pentanediones. Suitable organobismuth catalyst include bismuth carboxylates. When MDI prepolymers are used, combinations of organoiron and organotin catalysts are preferred for their better cures. Organometallic catalysts are advantageously used in amounts ranging from 0.001 to 0.1, preferably from 0.005 to 0.05 parts

per 100 parts by weight of polyol. Tertiary amine catalyst are advantageously used in amounts ranging from 0.01 to 5, preferably from 0.1 to 2 parts per 100 parts by weight of polyol.

Suitable surfactants include the block copolymers of ethylene oxide, and silicone surfactants. Suitable silicone surfactants include those disclosed, for example, in U.S. Patent No. 4,483,894. Such surfactants are advantageously employed in amounts ranging from 0.01 to 2 parts per 100 parts by weight of polyol.

Suitable filler materials include finely particulate materials such as, for example, calcium carbonate, aluminum trihydrate, titanium dioxide, iron oxide and barium sulfate. The use of such fillers is preferred, as they improve certain physical properties and often impart improved flame resistance to the polyurethane. Such fillers are advantageously used in amount ranging from 5 to 300, preferably from 20 to 500, more preferably from 70 to 130 parts per 100 parts by weight of polyol. Chemically blown systems, and systems employing MDI or derivatives as the polyisocyanate, can have even higher filler loadings.

Other optional ingredients of the polyurethane-forming composition include, for example, pigments, colorants, antistatic agents, reinforcing fibers, antioxidants, preservatives and water scavengers.

In preparing polyurethane-backed textiles according to this invention, the individual components of the polyurethane-forming composition are mixed and applied as a layer of preferably uniform thickness onto

one surface of the textile. It is often preferable to pre-mix all components except the polyisocyanate (and blowing agent, when a gas is used) to form a formulated "B-side". This simplifies the metering and mixing of components at the time the polyurethane-forming composition is prepared. In preparing a frothed polyurethane backing, it is preferred to mix all components and then blend a gas into the mixture, using equipment such as an Oakes or Firestone foamer.

The polyurethane-forming composition is then applied to one surface of a textile before it cures to a tack-free state. The composition may be applied to the textile before any significant curing takes place, using equipment such as a doctor knife, air knife or extruder to apply and gauge the layer. Alternatively, the composition may be formed into a layer on a moving belt or other suitable apparatus and partially cured, then married to the textile using equipment such as a double belt (double band) laminator or a moving belt with an applied foam cushion. After application and gauging, the layer is cured through the application of heat, such as by heating plates, a convection oven, an infrared oven, or other suitable apparatus. A cure temperature of from 100 to 170°C, for a period of from 1 to 60 minutes is suitable. Of course, the cure time is dependent on the temperature.

In one embodiment of the invention, the polyurethane-forming composition is applied to a textile which is already precoated with a certain polyurethane precoat layer. The use of this precoat layer is optional in this invention. The precoat layer is formed from a polyurethane precoat composition comprising, as reactive components, a polyether polyol,

a chain extender and a polyisocyanate, as described in U.S. Patent No. 4,296,159. Also useful are precoat formulations wherein the reactive components have an average functionality of from 1.95 to 2.02, as is described, for example, in U.S. Patent No. 4,696,849. The use of the precoat layer provides much greater latitude in both the processing conditions and the composition of the polyurethane-forming composition. In particular, the use of the precoat layer permits the practitioner a greater latitude in selecting the components of the polyurethane-forming composition. The precoat allows one to employ slightly higher functionality components in the precoat composition. The use of such higher functionalities tends to be detrimental when no precoat layer is used, due to the tendency of the coated textile to distort. However, the use of precoat layer prevents the polyurethane-forming composition from penetrating into the fibers of the textile, which substantially reduces the distortion associated with using higher functionality components. The use of slightly higher functionality components provides the added advantage of forming a lower tack polyurethane layer and providing better physical properties.

The amount of the polyurethane-forming composition used can vary widely, from 5 to 500 ounces per square yard, depending on the characteristics of the textile. For making carpet tile, for example, relatively high amounts of the composition are used. Preferably, from 10 to 200, more preferably from 30 to 120 ounces of polyurethane-forming composition are used per square yard of textile. When a precoat layer is

used, it advantageously weighs from 3 to 70, more preferably from 5 to 40 ounces per square yard.

Once the polyurethane-forming composition is applied to the textile, it is cured to a tack-free state. In order to further minimize distortion, it is preferred to handle the textile, at all times prior to final cure, such that mechanically induced stress is minimized. It is also preferred to cool the coated textile to a temperature of less than 40°C prior to rolling it up for storage or transportation, or cutting it into tiles or other shapes.

Methods for preparing polyurethane-backed textiles are disclosed, for example, in U.S. Patent Nos. 4,515,646, 4,595,436, 4,512,831, 4,485,894, 4,405,393, 4,278,482, 4.171,3954,022,941, 3,895,149 3,862,879, 3,849,156, 3,821,130 and 3,821,067.

In some applications, it is desirable to coat both sides of the textile with polyurethane, as in preparing carpet underlayment. This is easily done by coating the textile, then turning it over and coating the other side.

Multiple layers of polyurethane can, of course, be applied to the textile according to this invention. In addition, multiple layers of carpet or scrim materials can be employed. In particular, a non-woven scrim may be added to the textile as a bottom release layer to facilitate easy release from glue down installations.

The textile useful herein includes, for example, broadloom carpet tile, automotive carpet, fabrics for automotive trim, paneling and trunk liners,

synthetic playing surfaces, woven or non-woven polymeric scrims, tennis ball covers, drapery fabrics and wall covering fabric.

The polyurethane-backed textiles produced according to this invention are useful, for example, as attached cushion carpet, wall coverings, tennis balls, carpet underlayment and carpet tile.

The following examples are provided to illustrate the invention but are not intended to limit the scope thereof. All parts and percentages are by weight unless otherwise indicated.

Example 1

Polyurethane forming composition Sample No. 1 and Comparative Sample A were used in this example to prepare polyurethane-backed carpet. The blowing agent was air in both cases. The make-up of these compositions is provided in Table 1.

TABLE 1

| Component | Parts by Weight Sample No. 1 | Comp. Sample A* |
|---|---|---|
| Polyol Blend A[1] | 90 | 0 |
| Polyol B[2] | 0 | 90 |
| Diethylene glycol | 10 | 10 |
| Aluminum trihydrate | 40 | 40 |
| Calcium Carbonate | 40 | 40 |
| Organotin catalyst A[3] | 0.08 | 0.08 |
| Silicone surfactant | 0.125 | 0.125 |
| TDI prepolymer[4] | 38.9 | 40.3 |

[1]A 50/50 blend of a 2000 equivalent weight, 19% ethylene oxide-capped poly(propylene oxide) having a nominal functionality of 2 and an actual functionality of 1.75 and a 2000 equivalent weight, 17% ethylene oxide-cappped poly(propylene oxide) having a nominal functionality of 3 and an actual functionality of 2.26.

[2]A 1600 equivalent weight, 14% ethylene oxide capped poly(propylene oxide) having a nominal functionality of 3 and an actual functionality of 2.48.

[3]di(n-butyl)tinbis(isooctylmercaptacetate)

[4]A 26.8% -NCO prepolymer of TDI and a blend of a 200 molecular weight poly(ethylene oxide) diol and an adduct of glycerine and 3 moles of propylene oxide.

*Not an example of this invention.

The components were blended and frothed with air in an Oakes foamer to a weight of 440-450 g/quart (400-410 kg/$m^3$). The froth had a gel time of 3.1 minutes. The temperature of the froth was approximately (30°C) 88°F as it was obtained from the mixer. The pressure required to generate the froth was 110 psi (760 kPa) for Sample No. 1 and 114 psi (785 kPa) for Comparative Sample A. The frothed mixture was then applied to the reverse side of an 18.3 ounce (0.5 kg) space dyed level loop carpet having a nylon fabric tufted through a woven polypropylene primary, known commercially as Toronado, gauged to a coating weight as indicated in Table 2 following, and cured at 120°C for 8 minutes. The physical properties were determined on a portion of the backing which has been delaminated from the carpet.

**TABLE 2**

| Properties | Sample No. 1 | Comp. Sample A* |
|---|---|---|
| Coating Weight, oz/yd$^2$ (kg/m$^2$) | 43 (1.5) | 71 (2.4) |
| Density, lb/ft$^3$ (kg/m$^3$) | 20 (320) | 19 (304) |
| Penetration Wt., oz/yd$^2$ (kg/m$^2$) | 8.5 (0.3) | 16 (0.5) |
| Thickness, mils (mm) | 193 (4.9) | 234 (5.9) |
| Compression set, 50%, % | 3.7 | 3.1 |
| Tensile Strength, psi (MPa) | 116 (0.8) | 94 (0.65) |
| Elongation, % | 125 | 65 |
| Tear Strength, pli (N/m) | 9.0 (1575) | 6.0 (1050) |
| Bend Splits, min | 3+ | 3+ |
| Resiliency, % | 44 | 43 |
| 25% ILD | 24 | 29 |
| Cell count**, /mm$^2$ | 77 | 63 |

*-Not an example of this invention.

**-Determined from a foam sample doctored onto a flat plate.

As can be seen from the data in Table 2, the backing made according to this invention had significantly better tensile, elongation and tear properties than the control, while maintaining substantially equivalent properties in other respects. The ILD value was somewhat reduced in Sample No. 1, as is expected in a less crosslinked polyurethane, but was still sufficient to provide a high quality product.

The higher cell count obtained with this invention indicates that this composition froths more easily and uniformly than the control composition.

Example 2

Polyurethane-forming composition Sample No. 2 and Comparative Samples B and C were prepared from components as indicated in Table 3 following. The blowing agent in all cases was air.

TABLE 3

| Component | Parts by Weight | | |
|---|---|---|---|
| | Sample No. 2 | Comp. Samp. B* | Comp. Samp. C* |
| Polyol Blend A[1] | 90 | 0 | 0 |
| Polyol B[2] | 0 | 90 | 0 |
| Polyol C3 | 0 | 0 | 90 |
| Diethylene glycol | 10 | 10 | 10 |
| Aluminum trihydrate | 40 | 40 | 40 |
| Calcium Carbonate | 40 | 40 | 40 |
| Organoiron Catalyst[4] | 1 | 1 | 1 |
| Organotin catalyst B[5] | 0.01 | 0.01 | 0 |
| Silicone surfactant | 0.125 | 0.125 | 0.125 |
| MDI blend[6] | 34.4 | 35.7 | 34.3 |

*-Not an example of this invention.

[1-2]See notes 1-2 in Table 1.

[3]A 2000 equivalent weight, 17% ethylene oxide capped poly(propylene oxide) with an actual functionality of 2.26.

[4]A 2% solution of iron acetylacetonate in a 2000 molecular weight poly(propylene oxide) diol.

[5]di(n-isooctyl)tin bis (isooctylmercaptylacetate)

[6]A 60/40 blend of a 143 equivalent weight "liquid MDI" and a polymeric MDI having an o,p'- content of 12% and an average functionality of 2.18.

The compositions were used to prepare a polyurethane-backed carpet according to the same procedure described in Example 1. A significant processing parameter change was in the pressure required to froth the compositions. With the use of the MDI blend, the required pressure was 85 psi (586 kPa) for Sample No. 2, 80 psi (552 kPa) for Comparative Sample B and 96 psi (662 kPa) for Comparative Sample C. The use of MDI further permitted the use of lower curing temperatures. The properties of the resulting backing are as indicated in Table 4 following.

**TABLE 4**

| Properties | Sample No. 2 | Comp. Samp. B* | Comp. Samp. C* |
|---|---|---|---|
| Coating Weight, $oz/yd^2$ ($kg/m^2$) | 66 (2.2) | 65 (2.2) | 55 (1.9) |
| Density, $lb/ft^3$ ($kg/m^3$) | 21.5 (344) | 20 (320) | 20 (320) |
| Penetration Wt., $oz/yd^2$ ($kg/m^2$) | 13 (0.4) | 15 (0.5) | 12 (0.4) |
| Thickness, mils (mm) | 205 (5.2) | 213 (5.4) | 180 (4.6) |
| Compression set, 50%, % | 38.4 | 9.5 | 13.2 |
| Tensile Strength, psi (MPa) | 99 (0.68) | 84 (0.58) | 95 (.65) |
| Elongation, % | 75 | 50 | 53 |
| Tear Strength, pli (N/m) | 7.2 (1260) | 5.4 (945) | 5.6 (980) |
| Bend Splits, min | 3+ | 3+ | 3+ |
| Resiliency, % | 34 | 39 | 34 |
| 25% ILD, psi (kPa) | 30 (207) | 33 (228) | 30 (207) |
| Cell count**, $/mm^2$ | 68 | 44 | N.D. |

* - Not an example of this invention

**Determined from a foam sample doctored onto a flat plate.

As seen in Example 1, the process of this invention provided excellent tensile, elongation and tear properties, particularly as compared to those polyurethane-backed textiles made with the Comparative Samples. Using this process, a good quality MDI-based textile backing is obtained.

Example 3

Sample No. 1 and Comparative Sample A from Example 1 were repeated, except this time the polyurethane-forming compositions were frothed to a weight of 300 g/quart (270 kg/m$^3$). The physical properties of these foams are as indicated (as Sample No. 3 and Comparative Sample D, respectively) in Table 5 following.

TABLE 5

| Properties | Sample No. 3 | Comp. Sample D* |
|---|---|---|
| Density, lb/ft$^3$ (kg/m$^3$) | 15.5 (248) | 14.6 (234) |
| Thickness, mils (mm) | 213 (5.4) | 225 (5.7) |
| Compression set, 50%, % | 2.8 | 3.5 |
| Tensile Strength, psi (MPa) | 57.8 (0.4) | 54.5 (0.38) |
| Elongation, % | 93.7 | 51.9 |
| Tear Strength, pli (N/m) | 5.1 (893) | 4.9 (858) |
| Bend Splits, min | 3+ | 3+ |
| Resiliency, % | 44 | 45 |
| 25% ILD, psi (kPa) | 15.8 (109) | 19.3 (133) |

*-Not an example of this invention.

Improvement in tensile, elongation and tear are again seen.

Example 4

Sample No. 2 and Comparative Samples A and B from Example 1 are repeated, except this time the polyurethane forming compositions are frothed to a weight of 300 g/quart (284 mg/$m^3$). The physical properties of these foams are as indicated (as Sample No. 4 and Comparative Samples E and F) in Table 6 following.

TABLE 6

| Properties | Sample No. 4 | Comp. Samp. E* | Comp. Samp. F* |
|---|---|---|---|
| Density, lb/$ft^3$ ($kg/m^3$) | 15.9 (255) | 15.3 (245) | 14.9 (239) |
| Thickness, mils (mm) | 200 (5.1) | 164 (4.2) | 207 (5.3) |
| Compression set, 50%, % | 39.5 | 11.4 | 9.0 |
| Tensile Strength, psi (MPa) | 53.3 (0.37) | 48 (0.33) | 42 (0.29) |
| Elongation, % | 80.4 | 50.7 | 43.5 |
| Tear Strength, pli (N/m) | 4.1 (718) | 3.8 (665) | 3.5 (613) |
| Bend Splits, min | 3+ | 3+ | 3+ |
| Resiliency, % | 32 | 32 | 32 |
| 25% ILD, psi (kPa) | 16.4 (113) | 18.8 (130) | 17.7 (122) |

* Not an example of this invention.

Improvements in tensile, elongation and tear are again seen.

Example 5

Following the procedure of Example 2, polyurethane-forming composition Sample Nos. 5-8 were prepared from components as indicated in Table 7 following. The blowing agent in all cases was air.

TABLE 7

Parts by Weight

| Component | Sample No. 5 | Sample No. 6 | Sample No. 7 | Sample No. 8 |
|---|---|---|---|---|
| Polyol Blend A[1] | 90 | 90 | 90 | 92.3 |
| Diethylene glycol | 10 | 10 | 10 | 7.7 |
| Aluminum trihydrate | 40 | 40 | 40 | 50 |
| Calcium Carbonate | 40 | 40 | 40 | 47 |
| Organoiron Catalyst[4] | 0 | 1 | 1 | |
| Organotin catalyst A[5] | 0.01 | 0 | 0.01 | |
| Organotin catalyst B[6] | 0 | 0.01 | 0 | 0.018 |
| Silicone surfactant | 0.125 | 0.125 | 0.125 | 0.125 |
| MDI blend[7] | 37.5 | 0 | 0 | |
| MDI prepolymer A[8] | 0 | 40.6 | 0 | |
| MDI prepolymer B[9] | 0 | 0 | 42.3 | 0 |
| MDI Prepolymer C[10] | 0 | 0 | 0 | 34 |

[1-2]See notes [1-2] in Table 1.

[3]A 2000 equivalent weight, 17% ethylene oxide capped poly(propylene oxide) with an actual functionality of 2.26.

[4]A 2% solution of iron acetylacetonate in a 2000 molecular weight poly(propylene oxide) diol.

[5] di(n-isooctyl)tin bis (isooctylmercaptylacetate)

[6] di(n-butyl)tin bis (isooctylmercaptylacetate)

[7] A 60/40 blend of a 143 equivalent weight "liquid MDI" and a polymeric MDI having an o,p'- content of 12% and an average functionality of 2.18.

[8] A 52/48 mixture of an MDI prepolymer made by reacting 45 parts of a high o,p'-MDI with 7 parts tripropylene glycol and a 2.2 functional polymeric MDI having an o,p' content of 12%.

[9] A 60/40 by weight blend of a 181 equivalent weight MDI prepolymer and a polymeric MDI having an o,p'- content of 12% and an average functionality of about 2.18.

[10]A 50/50 by weight blend of a 181 equivalnet weight MDI prepolymer and a polymeric MDI havig an O,p'-content of 12% and an average functionality of 2.18

The resulting textile backings have properties as indicated in Table 8.

TABLE 8

| Properties | Sample No. 5 | Sample No. 6 | Sample No. 7 | Sample No. 8 |
|---|---|---|---|---|
| Density, lb/ft$^3$ (kg/m$^3$) | 20.6 (330) | 20.0 (320) | 21.9 (351) | 17.6 (282) |
| Compression set, 50%, % | 39.3 | 5.8 | 3.8 | 3.7 |
| Tensile Strength, psi (MPa) | 55.3 (0.38) | 49.3 (0.34) | 63.7 (0.44) | 32.1 (0.22) |
| Elongation, % | 90.3 | 54.8 | 95.6 | 93 |
| Tear Strength, pli (N/m) | 5.7 (998) | 5.5 (963) | 5.6 (980) | 2.7 (473) |
| Bend Splits, min | 3+ | 3+ | 3+ | 3+ |
| 25% ILD, psi (kPa) | 16.4 (113) | 18.8 (130) | 21.3 (147) | 9.6 (66) |

The backings have excellent properties. Of particular note are the greatly reduced compression sets of Sample Nos. 6, 7 and 8. This desirable property is attributable to the use of the MDI prepolymer. The use of the combination of organotin and organoiron catalysts in Sample Nos. 6 and 7 is further found to give improved cures when MDI prepolymers are used. Although a lesser amount of MDI prepolymer was used in Sample No. 8, its percent elongation was comparable to that of Sample No. 7.

The claims defining the invention are as follows:-

1. A process for preparing a polyurethane-backed textile, comprising the steps of

(A) applying to a surface of a textile a layer of a polyurethane-forming composition comprising

(1) a polyol based on a $C_3$-$C_8$ alkylene oxide, which polyol has an equivalent weight of from 1800 to 3000, and an internal poly(ethylene oxide) block or a terminal ethylene oxide cap constituting from 15 to 30 percent of the weight of the polyol, or mixture of such polyols, wherein said polyol or mixture thereof has an average functionality of from 1.8 to 2.2,

(2) from 5 to 25 parts, per 100 parts by weight of component (1), of a low equivalent weight compound having 2 active hydrogen-containing groups per molecule, and

(3) a polyisocyanate having an average functionality of from 1.9 to 2.4, in an amount to provide the composition with 0.9 to 1.25 isocyanate groups per active hydrogen-containing group, and

(4) an effective amount of a blowing agent and

(B) curing said polyurethane-forming composition to form a cellular polyurethane backing adherent to said textile.

2. A process as claimed in Claim 1 wherein component (1) comprises an ethylene oxide capped poly(propylene oxide) or mixture thereof and component (2) comprises an alkylene glycol or glycol ether.

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3. A process as claimed in Claim 2 wherein said polyurethane-forming composition further comprises a particulate, inorganic filler comprising aluminum trihydrate, calcium carbonate, barium sulfate or mixtures thereof.

4. A process as claimed in Claim 3 wherein component (A) comprises a mixture of a nominally trifunctional ethylene oxide-capped poly(propylene oxide) and a nominally difunctional ethylene oxide-capped poly(propylene oxide), said mixture having an average functionality of from 1.95 to 2.05.

5. A process as claimed in Claim 4 wherein said polyisocyanate comprises toluene diisocyanate or a prepolymer thereof with glycerine or trimethylolpropane or mixture thereof with an alkylene glycol or glycol ether, said prepolymer having an average functionality of from 2.03 to 2.2.

6. A process as claimed in Claim 4 wherein said polyisocyanate comprises a mixture of a liquid MDI having an equivalent weight of from 130 to 150 and a polymeric MDI, said mixture having an average functionality of from 2.03 to 2.2.

7. A process as claimed in Claim 4 wherein said textile is a carpet.

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8. An active hydrogen-containing composition comprising

(A) a polyol based on a $C_3$-$C_8$ alkylene oxide, which polyol has an equivalent weight of from 1800 to 3000 and an internal poly(ethylene oxide) block or a terminal ethylene oxide cap constituting from 15 to 30 percent of the weight of the polyol, or a mixture of such polyols, wherein said polyol or mixture thereof has an average functionality of from 1.8 to 2.2,

(B) from 5 to 25 parts, per 100 parts by weight of component (1), of a low equivalent weight compound having 2 active hydrogen-containing groups per molecule, and

(C) from 5 to 500 parts, per 100 parts of component (A), of an inorganic, particulate filler.

9. A composition as claimed in Claim 8 wherein component (A) comprises an ethylene oxide capped poly(propylene oxide) or mixture thereof and component (B) comprises an alkylene glycol or glycol ether.

10. A composition as claimed in Claim 9 further comprising a particulate, inorganic filler comprising aluminum trihydrate, calcium carbonate, barium sulfate or mixtures thereof.

11. A composition as claimed in Claim 10 wherein component (A) comprises a mixture of a nominally trifunctional ethylene oxide capped poly(propylene oxide) and a nominally difunctional ethylene oxide capped poly(propylene oxide), said mixture having an average functionality of from 1.95 to 2.05.

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12. A process for preparing a polyurethane-backed textile, comprising the steps of

(A) applying to a surface of a carpet a layer of a polyurethane-forming composition comprising

(1) 92.3 parts by weight of a 50/50 by weight blend of a 2000 equivalent weight, 19 percent ethylene oxide-capped poly(propylene oxide) having a nominal functionality of 2 and an actual functionality of 1.75 and a 2000 equivalent weight, 17 percent ethylene oxide-capped poly(propylene oxide) having a nominal functionality of 3 and an actual functionality of 2.26

(2) 7.7 parts by weight of diethylene glycol,

(3) 50 parts by weight aluminum trihydrate,

(4) 47 parts by weight calcium carbonate,

(5) 0.018 part by weight di(n-butyl)tin bis (isooctylmercaptylacetate),

(6) 0.125 part by weight silicone surfactant, and

(7) 34 parts by weight of a 50/50 by weight blend of a 181 equivalent weight MDI prepolymer and a polymeric MDI having an o,p' - content of 12 percent and an average functionality of 2.18, and

(8) an effective amount of a blowing agent,

(B) curing said polyurethane-forming composition to form a cellular polyurethane backing adherent to said carpet.

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13. A process according to Claim 8 substantially as hereinbefore described with reference to any one of the examples.

14. An active hydrogen-containing composition according to Claim 8 substantially as hereinbefore described with reference to any one of the examples.

15. A process according to Claim 12 substantially as hereinbefore described with reference to any one of the examples.

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