

MOISTURE CURABLE ADHESIVE COMPOSITION

Related Application

[0001] This application is a continuation-in-part of U.S. Application Serial No. 10/075,203 filed on February 14, 2002 and is hereby incorporated by reference herein.

Field of the Invention

[0002] The present invention relates to a moisture curable adhesive composition. The present invention is further directed to a method of joining two adherends.

BACKGROUND

[0003] Adhesives are commonly used to join or fasten two or more adherends. Adherends are considered as being any two or more materials, or pieces of material that are being joined together, including wood, metals, paper, ceramics, stone, glass, concrete, etc. Adhesives used for these purposes are based on a wide range of technologies, including elastomer/solvent/resin mixtures, epoxies, latexes, polyurethanes, silicones, cyanoacrylates, acrylics, hot melts, and others. These adhesives each have a number of drawbacks.

[0004] Adhesives based on elastomer/solvent/resin mixtures, henceforth referred to as rubber-based adhesives, contain solvents which are toxic and, most often, flammable. Despite these hazards, rubber-based adhesives are popular due to their ease of use. Epoxy, acrylic, some polyurethane, and some silicone adhesives are formulated into two-component systems consisting of a base containing monomers, oligomers, and polymers, and a hardener or curative component, consisting of catalysts and curing agents. These systems require accurate measuring and thorough mixing to develop the properties that make them useful as adhesives. The individual raw materials in two-component adhesive products, monomers, oligomers, catalysts, and curing agents, can be toxic and often allergenic. Latex adhesives are low toxicity, non-flammable materials, which harden

through evaporation of water. While effective in many applications, latex adhesives do not perform well in low temperature environments, on metals, on many plastics, and in wet or humid environments. In conditions of low temperature or high humidity, the drying time of latex adhesives can be greatly extended, sometimes for days. On the other hand, hot melt adhesives, being thermoplastic by definition, lose strength in warm conditions, limiting the applications in which they can be used. Additionally, hot melt adhesives require a source of heat for application, which further limits their use. These heat sources and the application of hot adhesive present a safety (burn) hazard to the user. Single component adhesives, such as polyurethanes and silicones, cure by reaction with moisture in air or on the adherends. Polyurethanes develop tack slowly, requiring that the adherends be held or fixtured in place (bracing or taping) until the adhesive has cured enough to hold them in place without being held. The same applies for single component silicone adhesives. Further, in a portion of the population, allergic reactions can result from exposure to polyurethane adhesives. In addition to the problems listed above, solvent and water based adhesives generally exhibit a high degree of shrinkage due to the volatile content of the adhesives.

[0005] The longevity and durability of adhesive bonding is dependent on a number of factors, including but not limited to surface properties of the adherends, the type and properties of the adhesive used in the bonding application, environmental conditions (temperature, humidity, etc.), the design of the adhesive joint, and the method of joining or assembling the adhesive and adherends together. High solids content adhesives solve some of the problems encountered with these other types of adhesives (i.e., shrinkage, amount of volatiles, etc), however the best of these adhesives present other drawbacks. For instance, European Patent 0 442 380 B 1 describes the use of a high solids content moisture curable adhesive with which two adherends can be bonded together. The patent description, however, indicates if the adhesive is applied too thickly, the adhesive cannot form a sufficient tacking force (tackiness), and becomes weak in the contact ability. It is therefore preferable that the adhesive be applied in the thickness of approximately 1mm or less so as to increase the tack.

[0006] The need to apply a thin layer of adhesive limits the applications an adhesive can be used for, and the means (i.e., roller, etc) for applying the adhesive. To apply thin layers of adhesives it is helpful to have an adhesive that can maintain its adhesive properties, and will

spread and flow across the substrate or adherend. If the viscosity of the adhesive is too high it can be difficult or impossible to apply a thin layer of adhesive (layers of 1mm or less). One of the factors which influences the viscosity of an adhesive is the amount of filler in the adhesive. In general, the higher the filler content the higher the viscosity. Filler content also affects the tack of the adhesive. At higher filler contents, the tack is lower. Therefore, to get a high solids content adhesive with sufficient tack and a low enough viscosity to spread thinly it has been necessary to use formulations with relatively low filler content. Further, it has been difficult to even add filler to high solids content clear adhesives since most filler including clear fillers will either block or refract light passing through an adhesive making the adhesive appear unclear. There is, however, a great need to increase the filler content of an adhesive since a higher filler content lowers the cost of an adhesive and reduces the environmental impact of manufacturing the adhesive.

[0007] Another problem associated with most good adhesives has been the repositionability of the adherends bonded by the adhesives. Conventionally, once an adhesive had developed enough tack to bond two adherends without clamps or some other type of temporary fasteners, then it was difficult to reposition the adhesive after contacting the adherends after more than a couple minutes. A need therefore exists for an adhesive, which develops tack rapidly so the two adherends to be bonded by the adhesive can be joined and held together without the use of clamps after a relatively short period of time, but where after joining the two adherends can be repositioned with respect to one another for a relatively long period of time.

[0008] It is, therefore, two of the objects of the present invention among the many objects of this invention to solve these problems.

SUMMARY

[0009] The present invention is directed to a moisture curable adhesive composition that includes, based on the total weight of the moisture curable adhesive composition, from about 5% to about 98% by weight polymer which contains a reactive silicone end group, and from about 0.01% to about 4% catalyst. The moisture curable adhesive composition optionally includes from about 0.01% to about 10% by weight dehydrating agent, based on the total weight of the moisture curable adhesive composition, to maintain a stable adhesive composition prior to reaction with moisture.

[0010] Suitable polymers include those which can contain the reactive silicon end groups. They include, but are not limited to, any polyalkyl oxide, including but not limited to, polyethers; any polyalkane, polyalkenes, and polyalkynes; substituted alkyl monomers, such as, for example, styrene; acrylics; any polymer or copolymer that can be prepared with the silyl (reactive silicon) end groups; and combinations thereof. Suitable reactive silicon end groups include but are not limited to: triethoxysilanes, methyldiethoxysilanes, trisilanols, any alkoxysilane, substituted silanes, multi-silanols, and combinations thereof. Examples of catalysts which may be used include, but are not limited to, tertiary amine catalysts, tin catalysts, lead catalysts, bismuth catalysts and mixtures thereof.

[0011] Also, there are no undesirable volatiles associated with the adhesive composition herein.

[0012] In another embodiment of the invention, the moisture curable adhesive composition includes, based on the total weight of the composition, from about 5% to about 95% by weight polymer having a reactive silicone end group, from about 0.01% to about 4% by weight catalyst, and from about 3% to about 35% diluent. Whereas conventional solvent-based adhesives take up to 20 to 30 minutes to achieve initial tack, the moisture curable adhesive has been surprisingly found to develop initial tack immediately or nearly immediately upon exposure to the air having a moisture content greater than about 20% relative humidity. The presence of a diluent improves flow, and the moisture curable adhesive of the present invention has a viscosity that preferably ranges from about 1,000 centipoise (cps) to about 500,000 cps and is advantageous for use in applications requiring high flowability.

[0013] The type of polymer having a reactive silicone end group, the reactive silicone end group and the catalyst, all of which are described above, can be used. Suitable diluents include, but are not limited to: polyglycols, for example, polypropylene glycol; mineral oils, such as white mineral oil and petroleum oil; vegetable oils, such as castor oil, corn oil, peanut oil; tall oil fatty acids, monofunctional polyether polymers; phthalate plasticisers, such as butyl benzyl phthalate, and diisodecyl phthalate; and mixtures thereof. Also, the moisture curable adhesive composition herein can optionally include a dehydrating agent, filler and an adhesion promoter.

[0014] In another embodiment of the invention, the moisture curable adhesive composition includes, based on the total weight of the composition, from about 15% to about 95% by weight polymer having a reactive silicone end group, from about 0.01% to about 4% by weight catalyst, and from about 5% to about 85% filler. The type of polymer having a reactive silicone end group, the reactive silicone end group and the catalyst, all of which are described above, can be used. Whereas conventional adhesive compositions are typically limited by the amount of filler loading, the moisture curable adhesive herein has excellent physical properties, such as elongation, tack, and shear strength, over a broad range of filler loading. In addition, the moisture curable adhesive composition herein can optionally include from about 0.01% to about 5% by weight a dehydrating agent, pigments, and from about 0.01% to about 10% by weight adhesion promoter.

[0015] In yet another embodiment of the invention, the moisture curable adhesive composition contains up to about 40% by weight filler, based on the total weight of the composition, to achieve an adhesive composition that is clear or substantially clear. Whereas fillers tend to cloud conventional adhesive compositions at relatively low filler loadings, it has been found, surprisingly, that a filler, such as, for example, a fumed silica which has a surface area that is less than about 250m²/gram can be used at substantially higher level loadings to achieve an adhesive composition that retains clarity.

[0016] The present invention is further directed to the method of making a moisture curable adhesive composition and a method of joining two adherends.

[0017] Additional features and advantages of the invention will be set forth in the detailed description which follows. It is to be understood that both the foregoing general description and the following detailed description are merely exemplary of the invention, and are intended to provide an overview or framework for understanding the nature and character of the invention as it is claimed.

DETAILED DESCRIPTION

Moisture Curable Adhesive Composition

[0018] The present invention is directed to a moisture curable adhesive composition that includes, based on the total weight of the moisture curable adhesive composition from about 5% to about 98%, preferably from about 10% to about 90%, and more preferably, from

about 20 to about 85 %, and even more preferably, from about 25% to about 75% by weight polymer having a reactive silicone end group; and from about 0.01% to about 4%, preferably from about 0.01 to about 3%, and more preferably, from about 0.3% to about 2% by weight of a catalyst.

[0019] The meaning of “polymer” as used throughout herein includes polymers and copolymers. Suitable polymers include, but are not limited to, those which can contain the reactive silicon end groups, including but not limited to: any polyalkyl oxide, including but not limited to polyethers; any polyalkane, polyalkene, or polyalkyne; substituted alkyl monomers, such as, for example, styrene; acrylics; any polymer or copolymer that can be prepared with the silyl (reactive silicon) end groups; and combinations thereof. Suitable reactive silicon end groups include but are not limited to: triethoxysilanes, methyldiethoxysilanes, trisilanols, any alkoxysilane, substituted silanes, multi-silanols, and combinations thereof. Examples of catalysts which may be used include, but are not limited to, tertiary amine catalysts, tin catalysts, lead catalysts, bismuth catalysts and mixtures thereof. Further details regarding catalysts are discussed below.

[0020] The moisture curable adhesive composition of the present invention contains substantially all solids, typically less than about 3% volatile components, and has excellent physical properties. The adhesive composition reacts upon exposure to moisture and has little or no shrinkage upon curing as contrasted with conventional adhesive compositions which experience substantial shrinkage due to evaporation of volatiles.

[0021] The moisture curable adhesive composition optionally includes from about 0.01% to about 10%, preferably from about 0.01% to about 5%, and more preferably, from about 0.01% to about 2% by weight of a dehydrating agent, based on the total weight of the moisture curable adhesive composition. A dehydrating agent is preferred to maintain a stable adhesive composition prior to reaction with moisture. The moisture curable adhesive composition preferably includes from about 0.01% to about 5 %, preferably from about 0.4% to about 3%, and more preferably, from about 0.5% to about 2% by weight of an adhesion promoter to increase the bond adhesive strength of the adhesive composition.

[0022] Depending on the packaging of the adhesive, the application for the adhesive, and the method of applying the adhesive a wide range of viscosities may be necessary. Preferably the adhesive composition has a viscosity as measured by ASTM D1084-88 Test

Method B from the container in which it is stored, of from about 1,000 to about 500,000 centipoise, more preferably from about 1,000 to about 300,000 centipoise, and more preferably from about 1,000 to about 200,000 centipoise.

[0023] In another embodiment of the invention, the moisture curable adhesive composition includes polymer having a reactive silicone end group and catalyst as described above, and also includes diluent. It has been found that the presence of a diluent in low viscosity moisture curable adhesive formulations, that is, less than about 10,000 centipoise and preferably less than about 3,000 centipoise improves the flow of the moisture curable adhesive composition and also improves the tack. In this embodiment the moisture curable adhesive composition includes, based on the total weight of the composition, from about 5% to about 95%, preferably, from about 10% to about 90%, even more preferably, from about 20% to about 85% by weight polymer having a reactive silicone end group; from about 0.01% to about 4%, preferably from about 0.01% to about 3% by weight, preferably from about 0.3% to about 3% by weight catalyst; and from about 3% to about 35%, preferably from about 7% to about 25%, preferably from about 10% to about 18%, and even more preferably from about 12% to about 16% diluent.

[0024] Whereas conventional solvent-based adhesives take up to 20 to 30 minutes to achieve tack, surprisingly the moisture curable adhesive has been found to develop tack in less than about 20 minutes, preferably less than about 10 minutes, more preferably less than about one minute, and even more preferably immediately, or nearly immediately, upon exposure to the air having moisture content greater than about 20% relative humidity, preferably greater than about 25% relative humidity, and even more preferably less than about 30% relative humidity. Tack is herein defined according to ASTM D 907 as the property of an adhesive that enables it to form a bond of measurable strength immediately after adhesive and an adherend are brought into contact under low pressure. Tack can be measured by the Rolling Ball Tack Test according to ASTM D 3121.

[0025] The presence of a diluent allows for adjustment of viscosity and tailoring of flow properties for various applications. The viscosity of the moisture curable adhesive composition depends on both the amount and the viscosity of the diluent. The viscosity of the diluent is preferably less than about 10,000 cps, more preferably less than about 8,000 cps, even more preferably, from about 2,000 cps to about 6,000 cps. The viscosity of the moisture curable adhesive composition can range from about 1,000 cps to about 500,000

cps, preferably, from about 1,000 cps to about 200,000, more preferably, from about 1,000 cps to about 100,000 cps. The moisture curable adhesive composition can be formulated to be self-leveling and is advantageous for use in applications requiring high flowability.

“Self-leveling” means that the moisture curable adhesive composition once placed in contact to an adherend will readily flow on the surface of the adherend to achieve a substantially even thickness.

[0026] The type of polymer having a reactive silicone end group, the reactive silicone end group and the catalyst, all of which are described above, can be used. Suitable diluents include, but are not limited to: polyglycols, for example, polypropylene glycol; mineral oils, such as, for example, white mineral oil and petroleum oil; vegetable oils, such as, for example, castor oil, corn oil, and peanut oil; tall oil fatty acids, monofunctional polyether polymers; phthalate plasticisers, such as, butyl benzyl phthalate, and diisodecyl phthalate; and mixtures thereof. Also, the moisture curable adhesive composition herein can optionally include a dehydrating agent, filler, and an adhesion promoter.

[0027] The moisture curable adhesive composition containing diluent can also optionally contain a dehydrating agent, an adhesion promoter, or both. The moisture curable adhesive composition herein can include from about 0.01% to about 10%, preferably from about 0.01% to about 5 %, and more preferably, from about 0.01% to about 2% by weight of a dehydrating agent, based on the total weight of the moisture curable adhesive composition, and from about 0.01% to about 5%, preferably from about 0.4% to about 3%, and more preferably, from about 0.5% to about 2% by weight of an adhesion promoter. For a given desired adhesive strength, less adhesion promoter will be required in the moisture curable adhesive composition as compared to the moisture curable adhesive embodiment that does not contain a diluent. Suitable dehydrating agents and adhesion promoters are well known in the art and are described below.

[0028] In another embodiment of the invention, any of the moisture curable adhesive composition of the present invention can further include filler. It has been found that relatively high levels of filler can be used to improve the strength of the adhesive composition while also maintaining the tack and viscosity characteristics of the adhesive. In one embodiment of the invention, the moisture curable adhesive composition includes, based on the total weight of the moisture curable adhesive composition: and from about 5% to about 95%, and preferably, from about 15% to about 85%, and more preferably from

about 25% to about 75% by weight polymer having a reactive silicone end group; from about 0.01% to about 4%, preferably, from about 0.01% to about 3%, and more preferably from about 0.2% to about 2% by weight catalyst; and from about 5% to about 85%, preferably from about 10% to about 40%, and more preferably from about 15% to about 40% filler. The type of polymer having a reactive silicone end group, the reactive silicone end group and the catalyst, all of which are described above, can be used. Whereas conventional adhesive compositions are typically limited by the amount of filler loading, the moisture curable adhesive herein has excellent physical properties, including weathering and retention of clarity, shear strength to a range of surfaces, and initial tack for a broad range of filler loading.

[0029] If clarity of the moisture curable adhesive composition is not necessary or desired, then the moisture curable adhesive herein contains a relatively high level of filler. It has been found, surprisingly, that the viscosity of the moisture curable adhesive composition retains good viscosity, from about 1,000 cps to about 500,000 cps, in addition to achieving excellent shear strength and tensile strength. The moisture curable adhesive composition having a high filler loading has also been found to exhibit good paintability. In one embodiment the moisture curable adhesive composition includes, from about 5% to about 95%, preferably 10% to about 85%, preferably, from about 20% to about 85%, and more preferably from about 25% to about 75% by weight polymer having a reactive silicone end group, from about 0.01% to about 4%, preferably, from about 0.01% to about 3%, and more preferably from about 0.2% to about 2% by weight catalyst, from about 20% to about 85%; preferably, from about 35% to about 85%; more preferably from about 43% to about 85%; and even more preferably from about 60% to about 85% by weight filler, based on the total weight of the moisture curable adhesive composition.

[0030] A wide range of fillers can be used and include, but are not limited to, zinc oxide, reinforcing carbon black, non-reinforcing carbon black, semi-reinforcing carbon black, white carbon, expanded graphite powder, powdery graphite, crystalline silica; molten silica; silicates; chalk; calcium carbonate including limestone; talc; mica; alumina; aluminum hydroxide; zirconia; titanium dioxide; wollastonite; feldspar; aluminum silicates; solid and hollow ceramic microspheres hollow spheres of ceramic or plastic; metal powders and microbeads; wood flour; dolomite; organic pigments inorganic pigments, fumed amorphous silica including hydrophilic and hydrophobic types, glass microbeads, glass fibers, clear

polymer fibers, clear polymer microbeads, clear polymer powders, and combinations thereof. An example of a suitable filler is calcium carbonate sold under the tradename Microwhite 100 by Imerys of Roswell, GA.

[0031] In another embodiment of the invention it has been found, surprisingly, that the moisture curable adhesive composition can maintain its clarity at filler level of up to about 40% by weight, based on the total weight of the moisture curable adhesive composition. By “clear” it is meant that the moisture curable adhesive is clear according to the Standard Test Method for Clarity and Cleanness of Paint and Ink Liquids as measured by ASTM D 2090 which is a test method under the jurisdiction of ASTM Committee D-1 on Paint and Coatings, Materials, and Applications. According to test method ASTM D 2090, clarity is defined as a complete lack of any visible nonuniformity when viewed in mass, in bottles or test tubes, by strong transmitted light. It has also been found that the moisture curable adhesive composition exhibits excellent UV stability which is typically uncharacteristic of a clear adhesive composition.

[0032] Thus, in another embodiment the moisture curable adhesive composition contains up to about 40%, preferably from about 5% to about 30%, more preferably from about 5% to about 20%, and even more preferably from about 10% to about 15% filler to achieve an adhesive composition that is clear, or substantially clear, while also having excellent tack and viscosity stability. Whereas conventional fillers, such as, for example, calcium carbonate and titanium dioxide, tend to produce an adhesive composition with visible haze at relatively low filler loadings, such as, for example, 0.05% by weight or more filler, it has been found that use of a clear filler produces a moisture curable adhesive composition that is clear at substantially higher filler concentrations.

[0033] Preferably, the filler has an index of refraction that is within about 30% of that of the moisture curable adhesive composition, more preferably, within about 20%, even more preferably, within about 10%, and most preferably within about 5% of the moisture curable adhesive composition. For example, about a 1 mm thick coating of the moisture curable adhesive composition preferably transmits greater than about 60% of the visible spectrum of sunlight, more preferably greater than about 75%, and most preferably greater than about 90%.

[0034] Examples of fillers that may be used to produce a moisture curable adhesive composition that is clear or substantially clear include but are not limited to: fumed amorphous silica including hydrophilic and hydrophobic types, glass microbeads, glass fibers, clear polymer fibers, clear polymer microbeads, clear polymer powders, or combinations thereof. Preferably, the filler for a clear adhesive is a fumed amorphous silica including, but not limited to, for example, hydrophilic fumed silicas and hydrophobic fumed silicas. Examples of suitable fumed silicas are sold by the Wacker HDK of Munich, Germany under the tradename S13, V15, V15A, N20, N20P for hydrophilic fumed silicas and H15, H15P, H20, H30, H18, and H2000 for the hydrophobic fumed silicas. Another suitable filler for a clear adhesive is a fumed silica sold under the tradename Aerosil OX-50 by Degussa Corporation of Ridgefield Park, NJ.

[0035] To produce a moisture curable adhesive composition that is clear, or substantially clear, the filler particles preferably have an average surface area of less than about 250 m²/gram, preferably, less than about 150 m²/gram, more preferably, less than about 75 m²/gram, and even more preferably, less than about 65 m²/gram. It is preferred that the filler has a surface area of less than about 90 m²/gram, and even more preferably, less than about 65 m²/gram to achieve clarity be used at substantially higher level loading. A moisture curable adhesive composition herein preferably includes less than about 10%, and even more preferably, less than about 7% by weight of a filler having a surface area that is greater than about 90 m²/gram, otherwise, the viscosity is substantially increased and the moisture curable adhesive composition can exhibit haze. The average number particle size of the filler should range from about 30 nm to about 70 nm, preferably, from about 40 nm to about 60 nm, and even more preferably from about 45 nm to about 55 nm. For example, filler that has a surface area that ranges from about 35 m²/gram to about 65 m²/gram, and an average number particle size of about 40 nanometers, for example, is Aerosil OX-50 by Degussa Corporation of Ridgefield Park, NJ. The Examples below show moisture curable adhesive compositions containing fumed silica of various surface area and the excellent results after UV exposure in a QUV weatherometer.

[0036] In another embodiment the moisture curable adhesive composition has two or more fillers having different surface area. Filler that has a surface area of greater than about 65 m²/gram, and preferably, greater than about 90 m²/gram are used to retard or eliminate sag. The moisture curable adhesive composition preferably contains up to about 4%, more

preferably up to about 3%, and even more preferably up to about 1.5% by weight filler having a surface area greater than about 90 m²/gram. As filler having a surface area of about 90 m²/gram and greater increases viscosity and also effects the clarity of the moisture curable adhesive composition, one skilled in the art will be able to determine the proper concentration of each of the two or more fillers, depending upon the surface area of the fillers, to maintain clarity of the moisture curable adhesive composition.

[0037] The moisture curable adhesive composition of the embodiments described above optionally includes from about 0.01% to about 4 %, more preferably from about 0.5% to about 3%; and most preferably from about 1.0% to about 2.5% by weight of an anti-oxidant, based on the total weight of the moisture curable composition. In addition, the moisture curable adhesive composition optionally includes additional optional ingredients, including but not limited to, anti-oxidants, catalysts, lubricants, extenders, biocides, adhesion promoters, UV absorbers and stabilizers.

[0038] Another significant advantage of the invention herein is the substantially short time to achieve tack, and the capability of repositioning the adherend or adherends once the moisture curable adhesive composition has achieved tack.

[0039] In addition, the moisture curable adhesive can be used in low temperature applications and over a broad range of application temperatures. For good low temperature application and use it is desirable that the moisture curable adhesive have a low glass transition temperature. Preferably, the moisture curable adhesive composition has a glass transition temperature (T_g) of less than about 0°C, more preferably less than about -20 °C and most preferably less than about -40°C. In order to use the adhesive in a variety of environments, it is desirable that the moisture curable adhesive has a broad application temperature range. The application temperature range is the temperature wherein the adhesive can be applied and still cure and function at or near its optimal curing and physical properties. Preferably, the application temperature range is from about -20 to about 60°C, more preferably from about 0°C to about 45°C, and most preferably from about 5°C to about 38°C. The service temperature range is that range of temperatures in which the cured adhesive will maintain substantially all its strength, adhesion and elasticity. Preferably, substantially all of the adhesive's strength, adhesion and elasticity are greater than 80% of the adhesive's maximum strength, adhesion and elasticity over this temperature range.

Preferably, the service temperature range for the adhesives of this invention is from about -60°C to about 160°C.

Catalyst

[0040] The moisture curable adhesive composition herein includes, but is not limited to, one or more of the following catalysts: tertiary amine catalysts, tin catalysts, lead catalysts, bismuth catalysts and mixtures thereof. Tertiary amine catalysts include by way of example and not limitation include compounds having one tertiary nitrogen atom including triethylamine, N,N-dimethylcyclohexylamine, N-methylmorpholine, N-ethylmorpholine, N-(2-hydroxyethyl)morpholine, N,N-dimethyl-p-toluidine, β -(dimethylamino)propionitrile, N-methylpyrrolidone, N,N-dicyclohexylmethylamine, and the like; compounds having two tertiary nitrogen atoms including N,N,N',N'-tetramethylethylenediamine, N,N,N',N'-tetramethylpropane-1,3-diamine, N,N,N',N'-tetramethylhexane-1,6-diamine, bis(N,N-dimethylaminoethyl)ether, bis(2-dimethylaminoethyl)ether, ethylene glycol bis(3-dimethylaminopropyl) ether, N'-cyclohexyl-N,N-dimethylformamidine, N,N'-dimethylpiperazine, trimethylpiperazine, 1,2-peperidinoethane, bis(aminopropyl)piperazine, N-methyl-N'-(2-hydroxyethyl)piperazine, N-methyl-N'-(2-hydroxyethyl)piperazine, N-(N',N'-dimethylaminoethyl)morpholine, bis(morpholinoethyl)ether, bis(2,6-dimethylmorpholinoethyl)ether, 1,2-dimethylimidazole, N-methylimidazole, 1,4-diazine, diazabicyclo[2.2.2]-octane, 1,4-diazabicyclo[3.3.0] oct-4-ene, 1,5-diazabicyclo[4.3.0]non-5-ene, 1,8-diazabicyclo-[5.4.0]-undec-7-ene, phenolates thereof, octoates thereof, and the like; compounds having three tertiary nitrogen atoms including N,N,N',N'',N''-pentamethyldiethylenetriamine, N,N,N',N'',N''-pentamethyldipropylenetriamine, tetramethylguanidine, N-cyclohexyl-N',N',N'',N''-tetramethylguanidine, N-methyl-N'-(2-dimethylamino)ethylpiperazine, 1,5,7-triazabicyclo[4.4.0]dec-5-ene, and the like; and compounds having four nitrogen atoms including 1,1,4,7,10,10-hexamethyltriethylenetetramine, 1,3,5-tris(N,N-dimethylpropyl)hexadro-1,3,5-triazine, and the like.

[0041] Tin catalysts include by way of example, but not limitation, stannous octoate, dibutyltin diacetate, dibutyltin dilaurate, dibutyltin mercaptide, dibutyltin thiocarboxylate, dibutyltin dimaleate, dioctyltin mercaptide, dioctyltin thiocarboxylate, dibutyl tin oxide in diisooctyl phthalate, aliphatic organotin, and mixtures thereof.

[0042] Lead, bismuth and mercury catalysts include by way of example but not limitation lead 2-ethylhexanate, bismuth neodecanoate, phenylmercury propionate, and the like.

Additional Optional Ingredients

[0043] In addition to the above-described components, the moisture curable adhesive composition of the present invention can optionally include an additive or combination of additives. Suitable additives include by way of example, but not limitation, anti-oxidants, catalysts, lubricants, extenders, biocides, adhesion promoters, UV absorbers and stabilizers. Some additives can operate to control the polymerization process, thereby affecting the physical properties (e.g., modulus, glass transition temperature) of the polymerization product formed from the composition. Others can affect the integrity of the polymerization product of the composition (e.g., protect against de-polymerization, oxidative degradation or biological attack).

Dehydrating Agent

[0044] The moisture curable adhesive composition also includes a dehydrating agent. Examples of dehydrating agents include, but are not limited to, vinyl trimethoxysilane, any vinyl alkoxysilane, inorganic and organic zeolites, and the like. Preferably, the dehydrating agent is vinyl trimethoxysilane sold under the tradename Silquest A-171 by Witco OSI Specialties of Danbury, CT.

[0045] Adhesion Promoter

[0046] As mentioned above, the moisture curable adhesive composition may also include an adhesion promoter. Examples of adhesion promoters include, but are not limited to, N-(beta-aminoethyl)-gamma-aminopropyltrimethoxysilane, N-(beta-aminoethyl)-gamma-aminopropyldimethoxymethylsilane, gamma-glycidoxypentyltrimethoxysilane, any substituted silane where one or more of the substitutions contains a different functional group and the like. A preferred adhesion promoter is N-(beta-Aminoethyl)-gamma-aminopropyltrimethoxysilane sold under the tradename Silquest A-1120 by Witco OSI Specialties of Danbury, CT.

Anti-Oxidant

[0047] The moisture curable adhesive composition may also include an anti-oxidant. Examples of anti-oxidants include, but are not limited to, sterically hindered phenolics, substituted benzotriazole, hindered amine light stabilizer (HALS), thiodiethylene *bis*(3,5-di-

tert-butyl)-4- hydroxyhydrocinnamate, any other UV-absorber or free-radical scavenger, or combinations thereof. Preferably, the anti-oxidant can include, but is not limited to, sterically hindered phenolics sold under the tradename Irganox 1010 by Ciba Specialty Chemicals of Tarrytown, NY; substituted benzotriazole sold under the tradename Tinuvin 327 by Ciba Specialty Chemicals of Tarrytown, NY; hindered amine light stabilizer sold under the tradename Tinuvin 770 by Ciba Specialty Chemicals of Tarrytown, NY; and combinations thereof.

[0048] Method For Making Moisture Curable Adhesive Composition

[0049] The above components of the moisture curable adhesive composition can be mixed using conventional processing equipment and technology. Preferably, steps are taken during the processing of the moisture curable adhesive composition to minimize the pickup of moisture by the composition, and in some instances to reduce the moisture of the composition. Generally this involves heating the mixture and/or pulling a vacuum to remove the water, however, any other method known to those skilled in the art could be used to achieve acceptable moisture content. Preferably, the moisture curable adhesive composition herein has a moisture content of less than about 1000 ppm, more preferably less than about 800 ppm, and most preferably less than about 600 ppm as measured by ASTM Test Method D4672. Also preferably, the adhesive composition has moisture content greater than about 200 ppm as measured by ASTM Test Method D4672. Preferably, the moisture curable adhesive composition has a volatile content of less than about 10% by weight, more preferably less than about 5% by weight, even more preferably less than about 3% by weight, even more preferably less than about 1% by weight and most preferably less than about 0.5% by weight of the overall adhesive composition as measured by ASTM Test Method D1490.

[0050] In one embodiment of the present invention, a moisture curable adhesive composition is used to join at least two adherends (or at least two materials or pieces of material to be adhered). The adherends include, but are not limited to, paper, metal, ceramic, glass, textiles, cloth, plastic, rubber, foams, masonry, wood, leather, composites and the like. Further, the two or more adherends can be of the same or different materials. The moisture curable adhesive composition comprising a polymer including reactive silicon end groups is applied to at least one of two adherends. The adhesive is preferably a

one-part type, but may also be two or more parts. The adhesive can be applied to a single side, two sides, or multiple sides of the adherends similar to conventional adhesives. The adhesive can be applied by conventional methods such as using a person's hands/fingers, spatulas, rollers, trowels or sprayers, and dispensed in conventional packaging such as tubes, cans or containers. Preferably, the moisture curable adhesive composition is applied that is initially up to about 7 mm, more preferably greater than about 1 mm to about 5 mm, and more preferably from about 1.5 to about 4 mm in thickness. It is preferred that the moisture curable adhesive composition is applied over at least 25% of the adherend's surface that will be joined to the other adherend.

[0051] The surfaces of the adherends to be bonded may be maintained in non-contact with each other until the adhesive develops a sufficient tack to hold the adherends together as a result of exposure to atmospheric moisture. Preferably, the adherends are maintained in non-contact with respect to the other(s) for less than about 20 minutes, preferably for less than about 15 minutes, and more preferably for less than about 10 minutes, and even more preferably less than about 1 minute.

[0052] Once the adhesive develops a tack the adherends to be joined are first contacted forming a bond between them with the adhesive. Preferably, the tack is sufficient to temporarily prevent the adherends from pulling apart prior to the curing of the adhesive. Preferably, the adherends can be repositioned for some period of time prior to initial cure. The initial cure is defined as the time during which the assembly can be handled, i.e. moved, without degrading the bond. After this time, the substrates can no longer be repositioned without permanently degrading the bond strength. In the case of a bond where one substrate is immovable, i.e. a wall, it is the point at which bracing is no longer necessary to hold the substrates together. Preferably, at least two adherends can be repositioned with respect to each other for more than about 5 minutes after they are first contacted, more preferably for more than about 10 minutes, even more preferably for more than about 15 minutes, even more preferably for more than about 20 minutes, even most preferably for more than about 30 minutes. Also preferably, the bond between the at least two adherends is cured sufficiently to prevent further degradation of such bond after a reasonable period of time. The inventors, preferably, consider a 90% cure to be sufficient to make it reasonably difficult to degrade the bond through use or misuse of the bonded adherends. A 90% cure is defined as when about 90% of the available reactive silicon end groups have reacted. At 23°C and 50 % relative humidity, preferably the adhesive is about

90% cured in less than about 24 hours, more preferably is about 90% cured in less than about 16 hours, even more preferably is about 90% cured in less than about 12 hours and most preferably is about 90% cured in less than about 8 hours.

The foregoing detailed description is given for clearness of understanding only, and no unnecessary limitations should be understood therefrom. Hence, numerous modifications and changes can be made by those skilled in the art without departing from the spirit and scope of the invention. The following examples will serve to further typify the nature of the invention, but should not be construed as a limitation on the scope thereof, which is defined solely by the appended claims.

WORKING EXAMPLES

Examples 1 - 3

[0053] In Examples 1-3, moisture-curable adhesive compositions, listed in Table 1 below, were generally prepared by the following procedure: a translucent, low viscosity adhesive was made by mixing a polypropylene oxide with a methyldimethoxysilyl functional group sold under the trade name MS Polymer S303H by Kaneka of Osaka, Japan in a low speed Sigma Blade Lab Mixer from Teledyne Readco of York, PA keeping the mixer covered to minimize moisture exposure. The mixer was started on a low speed and the following materials were added: one of three polypropylene glycols sold under the tradename Arcol Polyol PPG-2000, PPG-3025 and PPG-4000 by Bayer of Pittsburgh, PA, and a sterically hindered phenolic anti-oxidant sold under the tradename Irganox 1010 by Ciba Specialty Chemicals of Tarrytown, NY. The mixer was then sealed and heated to between 71°C - 77°C. While heating, a vacuum was applied to the batch in the mixer to continuously distill off any water in the raw materials.

[0054] The mixture was tested for water content after mixing at the target temperature to determine if the target level of less than 1000 ppm was obtained. Heating and vacuum were continued until the moisture content was below 1000ppm. Once this level was attained, the heat was shut off and the vacuum broken.

[0055] When the batch had cooled to below 50°C, a vinyl trimethoxysilane dehydrating agent sold under the tradename Silquest A-171 Silane by Witco OSI Specialties of Danbury, CT was added to the mixture and mixed at low speed for five minutes. Afterwards, N-(3-

trimethoxy-1,2-ethanediamine) silane adhesion promoter sold under the tradename Silquest A-1120 by Witco OSI Specialties of Danbury, CT was added to the mixture and mixed at low speed for 5 minutes. Afterwards, dibutyl tin oxide in diisooctyl phthalate catalyst sold under the tradename FomRez SUL-11A by Witco OSI Specialties of Danbury, CT was added to the mixture, a vacuum was drawn, and the mixture was mixed at low speed for five minutes. The mixture was then transferred to an air-tight container for further testing.

Table 1

<u>Raw Material</u>	<u>Ex. 1 (wt%)</u>	<u>Ex. 2 (wt%)</u>	<u>Ex. 3 (wt%)</u>
Kaneka S303 H Polymer	623 (74.264)	638.6	613 (74.3)
Kaneka MAX 601 Polymer	0	0	0
Arcol PPG-2000 Diluent	166 (19.788)	0.9	0
Arcol PPG-3025 Diluent	0	105.5	0
Arcol P-4000 Diluent	0	0.9	163.0
Santicizer 100 Plasticizer	0	60	0
Tinuvin 327 Antioxdant	0	0.9	0
Tinuvin 770 Light Stabilizer	0	0.9	0
Aerosil R972	0	60	0
Irganox 1010 Antioxidant	6.2 (7.39)	0.9	6.0
Silquest A-171 Silane (Dehydrating Agent)	12.5 (1.490)	14.7	12.3 (1.5)
Silquest A-1120 Silane (Adhesion Promoter)	18.7 (2.229)	21.3	18.4 (2.2)
FomRez SUL-11A Catalyst	12.5 (1.490)	14.7	12.3 (1.5)

Shear Strength

ASTM D 1002 (Modified) Note: The modification consists of using surfaces other than metal. All other parameters remain the same.

The viscosity of the composition of Example 1 (Brookfield Viscometer, T/B Spindle, 5 rpm at 23°C): 2400 cps. The adhesive of the composition from Example 1 was spread on one layer of canvas adherend and immediately covered with a second canvas adherend and the peel strengths were measured. The peel strengths of the five test samples were 4.7, 4.4, 4.5, 4.3, and 4.7 pounds per lineal inch (pli) with an average peel strength of 4.5 pli plus or minus 0.25 pli.

The adhesive composition of Example 1 was applied between formica and plywood adherends. The moisture curable adhesive composition was troweled on one surface only

(1/8" x 1/8") and then tested for adhesion. The formica/plywood assembly was placed in an oven and heated to 105°C for approximately 3 hours. No edge lifting was observed.

The composition of Example 2 was tested for shear strength on wood. According to ASTM D 1002, the shear strength was as follows: 147.6 psi after 24 hours from application, 198.5 psi after 48 hours, and 175.2 psi after 1 week.

The hardness of the adhesive composition of Example 2 was also tested. The Shore A Hardness was 32A after 24 hours from application, and was 37A after 48 hours.

<u>Shore A Hardness</u>	
24 hrs.	32
48 hrs.	37

The shear strength of the composition of Example 2 was tested on wood after 24 hrs, 48 hrs and 1 week. The results were as follows:

<u>Shear Strength (Wood):</u>	
24 hrs.	147.6 psi
48 hrs.	198.5 psi
1 week	175.2 psi

Examples 4 - 11

[0056] In Examples 4-11, moisture-curable adhesive compositions, listed in Table 2 below, were generally prepared by the following procedure: a clear adhesive was made by mixing polypropyleneoxide with a methyldimethoxysilyl functional group sold under the tradename MS Polymer S303H by Kaneka of Osaka, Japan in a low speed Sigma Blade Lab Mixer from Teledyne Readco of York, PA keeping the mixer covered to minimize moisture pickup. The Sigma Blade Lab Mixer was started on a low speed and the following were added: fumed amorphous silica filler with a surface area of 50 m²/gram sold under the tradename Aerosil OX-50 by Degussa Corporation of Ridgefield Park, NJ; a substituted benzotriazole anti-oxidant sold under the tradename of Tinuvin 327 by Ciba Specialty Chemicals of Tarrytown, NY; a hindered amine light stabilizer anti-oxidant sold under the tradename of Tinuvin 770 by Ciba Specialty Chemicals of Tarrytown, NY; and a sterically

hindered phenolic anti-oxidant sold under the tradename Irganox 1010 by Ciba Specialty Chemicals of Tarrytown, NY.

[0057] The mixer was then sealed and heated to between 71°C - 77 °C. While heating, a vacuum was pulled on the mixer to continuously distill off any water in the mixture. The mixture was tested after reaching the target temperature to determine whether the target moisture level of less than 1000 ppm was obtained. The heating and vacuum were continued until this target was achieved. Once attained, the heat was shut off and the vacuum broken.

[0058] Once the batch had cooled to less than 50°C, then a vinyl trimethoxysilane dehydrating agent sold under the tradename Silquest A-171 Silane by Witco OSI Specialties of Danbury, CT was added to the mixture and mixed at low speed for 5 minutes. Afterwards, N-(3-trimethoxy-1,2-ethanediamine) silane adhesion promoter sold under the tradename Silquest A-1120 by Witco OSI Specialties of Danbury, CT was added to the mixture and mixed at low speed for 5 minutes. Afterward, dibutyl tin oxide in diisooctyl phthalate catalyst sold under the tradename FomRez SUL-11A by Witco OSI Specialties of Danbury, CT was added to the mixture, a vacuum was drawn and the mixture was mixed at low speed for 5 minutes. The mixture was then transferred to air-tight containers for further testing.

Table 2

<u>Raw Material</u>	<u>Ex. 4 (wt%)</u>	<u>Ex. 5 (wt%)</u>	<u>Ex. 6 (wt%)</u>	<u>Ex. 7 (wt%)</u>	<u>Ex. 8 (wt%)</u>	<u>Ex. 9 (wt%)</u>	<u>Ex.10</u>	<u>Ex.11</u>
Kaneka S303 H Polymer	626 (67.2)	626 (67.2)	0	0	760 (84.9)	584	725.7	725.7
Kaneka MAX 601 Polymer	0	0	760 (76.3)	689 (70.3)	0	0	0	0
OX-50 Fumed Silica	243 (26.1)	203 (21.8)	150 (15.1)	204 (20.8)	0	0	30.0	10.0
Aerosil R 972 Fumed Silica	0	40 (4.29)	15 (1.51)	13.6 (1.39)	63.4 (7.08)	0	30.0	50.0
Aerosil 90 Fumed Silica	0	0	0	0	0	110 (14.7)	0	0
Tinuvin 327 Antioxdant	6.3 (.68)	6.3 (.68)	6 (.60)	5.4 (.55)	6.3 (.68)	1.7	0.9	0.9
Tinuvin P Light Stabilizer	6.3 (.68)	6.3 (.68)	0	0	0	0	0	0
Tinuvin 770 Light Stabilizer	6.3 (.68)	6.3 (.68)	6 (.60)	5.4	6.3	1.7	0.9	0.9
Irganox 1010 Antioxidant	6.3 (.68)	6.3 (.68)	6	5.4	6.3	1.7	0.9	0.9
Silquest A-171 Silane	12.5 (1.34)	12.5 (1.34)	15.2	13.8 (1.41)	15.2	14.4	14.7	14.7
Silquest A-1120 Silane	18.8 (2.02)	18.8 (2.02)	0	0	22.8 (2.55)	21.2	0	0
Silquest A-2120 Silane	0	0	22.8 (2.29)	29 (2.96)	0	0	0	0
FomRez SUL-11A Catalyst	12.5 (1.34)	12.5 (1.34)	15.2	13.8	15.2 (1.70)	2.8	14.7	14.7
Neostann U-220 Catalyst	0	0	0	0	0	10	0	0

*All weights in grams

The shear strength of the moisture curable adhesive composition of Example 4 was tested according to ASTM D 1002 and the results are as follows:

<u>Time</u>	<u>Wood</u>	<u>Polystyrene</u>	<u>PVC</u>	<u>ABS</u>
24 hours.	284.7	113.9	94.0	99.5
1 week	286.3	159.3	145.6	115.6

The moisture curable adhesive composition of Example 4 containing 27.5% by weight fumed silica (OX-50) was placed in a Q-UV Weatherometer. After 1,687 hrs, the adhesive composition showed only slight yellowing and cracking.

The shear strength of the composition of Example 5 containing 21.8% by weight of fumed silica filler was tested for shear strength after a 24 hour cure and after a 1 week cure on several different substrates. The results were as follows:

<u>Time</u>	<u>ABS</u>	<u>Polystyrene</u>	<u>PVC</u>	<u>Wood</u>	<u>Aluminum</u>
24 hours.	75.5	115.6	120.0	248.9	127.3
1 week	92.8	127.2	143.4	274.0	138.6

The composition of Example 9 was also tested for Q-UV. The composition of Example 9 containing 14.7% fumed silica having a surface area of about 90 meters squared per grams performed as follows:

100 hours:	No yellowing
150 hours:	Slight yellowing
938 hours:	Moderate yellowing

The shear strength of the composition of Example 9 was also measured on wood samples and the results are as follows:

24 hours:	255.0 psi, 50% wood failure
48 hours:	273.0 psi, 100% wood failure
1 week:	274.1 psi, 100% wood failure

UV Weathering:

An adhesive composition of Example 10 containing 3.7% by weight of fumed silica and one from the composition of Example 11 containing 1.2% by weight of fumed silica were tested

for weathering. These compositions were cast on aluminum and were allowed to cure for 24 hours at room temperature. The samples were then placed in the Q-UV Weatherometer. After 100 hours of UV exposure, it was noted that the composition of Example 10 having relatively less fumed silica, had started to yellow. In the composition of Example 11, there was slight yellowing after 100 hours of UV exposure. However, the results of the composition of Example 11 experienced slight yellowing after 160 hours in the weatherometer.

[0059] Samples from Examples 4 – 8 and 13 were tested for viscosity following the procedure established under ASTM D 1084-88 Test Method B to measure the viscosity of the mixture initially and 24 hours later and the measurements are listed below:

Viscosity Initial (centipoise)	Viscosity 24 hours later (centipoise)
4,000	48,000
72,000	464,000
16,000	200,000
48,000	784,000
128,000	200,000
80,000	250,000

[0060] The testing showed that the viscosity using a fumed amorphous silica was dependent on the surface area of the silica particles used. The viscosity of samples from Examples 2 and 4 were in an unusable range when measured at 24 hours. The testing of the sample from Example 6 also showed that high filler loading can be achieved without increasing the viscosity beyond the usable range of the adhesive.

Examples 12 and 13

[0061] In Examples 12-13, moisture-curable adhesive compositions, listed in Table 3 below, were generally prepared by the following procedure: a white adhesive was made by mixing a polypropyleneoxide with a methyldimethoxysilyl functional group sold under the tradename MS Polymer S303H by Kaneka of Osaka, Japan in a low speed Sigma Blade Lab Mixer from Teledyne Readco of York, PA keeping the mixer covered to minimize moisture pickup. The Sigma Blade Lab Mixer was started on a low speed and the following were added: a butyl benzl phthalate sold under the tradename Santicizer 160 by Solutia, Inc. of St. Louis, MO; a calcium carbonate sold under the tradename Wingdale White by Imerys of

Roswell, GA; a titanium dioxide whitener sold under the tradename Tiona RCL-9 by Millenium Inorganic Chemicals Inc. of Baltimore, MD; a fumed amorphous silica filler with a surface area of 200 m²/gram sold under the tradename Aerosil 200 by Degussa Corporation of Ridgefield Park, NJ; a substituted benzotriazole anti-oxidant sold under the tradename of Tinuvin 327 by Ciba Specialty Chemicals of Tarrytown, NY; a hindered amine light stabilizer anti-oxidant sold under the tradename of Tinuvin P by Ciba Specialty Chemicals of Tarrytown, NY; and a sterically hindered phenolic anti-oxidant sold under the tradename Irganox 1010 by Ciba Specialty Chemicals of Tarrytown, NY.

[0062] The mixer was then sealed and heated to between 71°C - 77°C. While heating, a vacuum was pulled on the mixer to continuously distill off any water in the mixture. The mixture was tested after reaching the target temperature to determine whether the target moisture level of less than 1000 ppm was obtained. The heating and vacuum were continued until this target was achieved. Once attained, the heat was shut off and the vacuum broken.

[0063] Once the batch had cooled to less than 50°C, then a vinyl trimethoxysilane dehydrating agent sold under the tradename Silquest A-171 Silane by Witco OSI Specialties of Danbury, CT was added to the mixture and mixed at low speed for 5 minutes. Afterwards, a N-(3-trimethoxy-1,2-ethanediamine) silane adhesion promoter sold under the tradename Silquest A-1120 by Witco OSI Specialties of Danbury, CT was added to the mixture and mixed at low speed for 5 minutes. Afterwards, a dibutyl tin oxide in diisooctyl phthalate catalyst sold under the tradename FomRez SUL-11A by Witco OSI Specialties of Danbury, CT was added to the mixture, a vacuum was drawn and the mixture was mixed at low speed for 5 minutes. The mixture was then transferred to an air-tight container for further testing.

Table 3

<u>Raw Material</u>	<u>Ex. 11</u>	<u>Ex. 10</u>
Kaneka S303 H Polymer	134	400
Kaneka MAX 601 Polymer	0	
Norsolene S105	122	0
Calcium Carbonate	625	0
Santicizer 100 Plasticizer	0	220
Wingdale White Cal. Carbonate	0	550
Tiona RCL-9 Titanium Dioxide	0	50

Tinuvin 327 Antioxdant	1.8	3
Tinuvin P Light Stabilizer	0	3
Tinuvin 770 Light Stabilizer	1.8	3
Irganox 1010 Antioxdant	6.2	0
Aerosil R972	0	6
Silquest A-171 Silane (Dehydrating Agent)	4.5	10
Silquest A-1120 Silane (Adhesion Promoter)	0	0
Silquest A-2120 Silane (Adhesion Promoter)	0	6
FomRez SUL-11A Catalyst	4.5	0

The viscosity of the adhesive composition of Example 12 was measured initially and after 24 hours. The initial viscosity (Brookfield T/D at 5 rpm) equal 332,000 cps. 24 hour viscosity equals 370,000 cps. The shear strength of the adhesive composition of Example 12 were as follows:

24 hours 181.2 psi

48 hours 188.2 psi

The shear strength resulted in substrate failure.

The sample is difficult to process due to the high viscosity of the formulation, but did provide moisture curable adhesive product with properties to make it a functional adhesive.

WHAT WE CLAIM IS:

1. A moisture curable adhesive composition comprising:
 - from about 15% to about 90% by weight polymer with a reactive silicone end group, based on the total weight of the composition;
 - from about 0.01% to about 4% by weight catalyst, based on the total weight of the moisture curable composition; and
 - from about 3% to about 25% diluent, based on the total weight of the moisture curable composition.
2. The moisture curable adhesive composition of claim 1 wherein the polymer is selected from the group consisting of: polyalkyl oxide, polyalkane, polyalkene, polyalkyne, substituted alkyl monomers, and mixtures thereof.
3. The moisture curable adhesive composition of claim 1 wherein the reactive silicone group is selected from the group consisting of: triethoxysilane, methyldiethoxysilane, trisilanol, alkoxysilane, substituted silane, multi-silanol, and combinations thereof.
4. The moisture curable composition of claim 1 further comprising:
 - from about 0.01% to about 5% by weight adhesion promoter, based on the total weight of the moisture curable composition.
5. The moisture curable composition of claim 4 further comprising:
 - from about 0.01% to about 10% by weight dehydrating agent, based on the total weight of the moisture curable composition.
6. The moisture curable adhesive composition of claim 1 wherein:
 - the polymer is selected from the group consisting of: polyalkyl oxide, polyalkane, polyalkene, polyalkyne, substituted alkyl monomers; and mixtures thereof; and
 - the reactive silicone group is selected from the group consisting of:
 - triethoxysilane, methyldiethoxysilane, trisilanol, alkoxysilane, substituted silane, multi-silanol, and combinations thereof.

7. The moisture curable adhesive composition of claim 6 wherein the diluent is selected from the group consisting of:
polyglycol, mineral oil, vegetable oil; tall oil fatty acid, monofunctional polyether polymer; phthalate plasticiser, and mixtures thereof.
8. The moisture curable adhesive of claim 7 wherein the diluent is a polyglycol.
9. The moisture curable composition of claim 1 wherein the viscosity ranges from about 1,000 cps to about 500,000 cps.
10. The moisture curable adhesive composition of claim 1 wherein the catalyst is selected from the group consisting of: tertiary amine catalysts, tin catalysts, lead catalysts, bismuth catalysts and mixtures thereof.
11. The moisture curable adhesive composition of claim 6 wherein the catalyst is a tin catalyst selected from the group consisting of: stannous octoate, dibutyltin diacetate, dibutyltin dilaurate, dibutyltin mercaptide, dibutyltin thiocaboxylate, dibutyltin dimaleate, dioctyltin mercaptide, dioctyltin thiocarboxylate, dibutyl tin oxide in diisooctyl phthalate, aliphatic organotin, and mixtures thereof.
12. The moisture curable adhesive composition of claim 1 wherein the catalyst is selected from the group consisting of tin catalyst, bismuth catalyst and mixtures thereof.
13. The moisture curable composition of claim 1 wherein the glass transition temperature of the moisture curable composition is less than about -20°C.
14. The moisture curable composition of claim 1 wherein the service temperature of the adhesive ranges from about -60°C to about 160°C.
15. The moisture curable composition of claim 1 wherein the moisture curable composition achieves tack in less than about 20 minutes after exposing the moisture curable composition to moisture in air of greater than about 20% relative humidity.

16. The moisture curable composition of claim 1 wherein the moisture curable composition achieves tack in less than about one minute upon exposing the moisture curable composition to moisture in air of greater than about 25% relative humidity.

17. The moisture curable composition of claim 11 wherein the moisture curable composition achieves tack in less than about 20 minutes after exposing the moisture curable composition to moisture of greater than about 25% relative humidity.

18. A moisture curable composition comprising:
from about 15% to about 90% by weight polymer with a reactive silicone end group, based on the total weight of the moisture curable adhesive composition;
from about 0.01% to about 4% by weight catalyst, based on the total weight of the moisture curable adhesive composition;
from about 0.01% to about 5% by weight adhesion promoter;
from about 0.01% to about 10% by weight dehydrating agent;
from about 3% to about 25% by weight diluent;
wherein the polymer is selected from the group consisting of: polyalkyl oxide, polyalkane, polyalkene, polyalkyne, substituted alkyl monomers, and mixtures thereof;
wherein the moisture curable adhesive composition of claim 1 wherein the reactive silicone group is selected from the group consisting of:
triethoxysilane, methyldiethoxysilane, trisilanol, alkoxysilane, substituted silane, multi-silanol, and combinations thereof.

19. The moisture curable composition of claim 18 wherein the diluent is polypropylene glycol.

20. The moisture curable adhesive composition of claim 19 wherein:
the polymer with a reactive silicone end group is polypropyleneoxide and the reactive silicone end group is methyldimethoxysilyl functional group; and
the catalyst is selected from the group consisting of tin catalyst, bismuth catalyst and mixtures thereof.

21. A method of joining a first adherend and a second adherend comprising the steps of:

applying a moisture curable adhesive comprising a polymer having a reactive silicon end group, a catalyst and diluent, to at least one adherend;

maintaining the first adherend and the second adherend in non-contact with each other for less than about 20 minutes until the adhesive begins to cure; and

placing a second adherend in contact with the moisture curable adhesive applied to the first adherend to form a bond between the two adherends.

22. The method of claim 21 wherein:

at least one of the first adherend and the second adherend can be repositioned within about 5 minutes of placing the second adherend in contact with the first adherend.

23. A moisture curable adhesive composition comprising:

from about 5% to about 90% by weight polymer with a reactive silicone end group, based on the total weight of the composition;

from about 0.01% to about 4% by weight catalyst, based on the total weight of the moisture curable composition; and

from about 5% to about 95% filler, based on the total weight of the moisture curable composition.

24. The moisture curable adhesive composition of claim 23 wherein:

the moisture curable adhesive composition comprises from about 40% to about 85% by weight filler, based on the total weight of the moisture curable adhesive composition; and

wherein the moisture curable adhesive composition has a viscosity that ranges from about 1,000 cps to about 500,000 cps.

25. The moisture curable adhesive composition of claim 24 wherein:

the filler is selected from the group consisting of :

zinc oxide, reinforcing carbon black, non-reinforcing carbon black, semi-reinforcing carbon black, white carbon, expanded graphite powder, powdery graphite, crystalline silica, molten silica, silicates, chalk, calcium carbonate, limestone, talc, mica, alumina, aluminum hydroxide, zirconia, titanium dioxide, wollastonite, feldspar, aluminum silicates, solid and hollow ceramic microspheres hollow spheres of ceramic or plastic, metal powders and microbeads, wood flour, dolomite, organic pigments inorganic pigments, fumed amorphous silica including hydrophilic and hydrophobic types, glass microbeads, glass fibers, clear polymer fibers, clear polymer microbeads, clear polymer powders, and combinations thereof.

26. The moisture curable adhesive composition of claim 25 wherein:

the polymer is selected from the group consisting of:

polyalkyl oxide, polyalkane, polyalkene, polyalkyne, substituted alkyl monomers, and mixtures thereof;

the reactive silicone group is selected from the group consisting of: polyalkyl oxide, polyalkane, polyalkene, polyalkyne, substituted alkyl monomers, and mixtures thereof;

wherein the moisture curable adhesive composition further comprises:

from about 0.01% to about 5% by weight adhesion promoter, based on the total weight of the moisture curable composition; and

from about 0.01% to about 10% by weight dehydrating agent, based on the total weight of the moisture curable composition.

27. The moisture curable adhesive composition of claim 23 wherein:
- the moisture curable adhesive composition comprises up to about 40% by weight filler, based on the total weight of the moisture curable adhesive composition; and
- the moisture curable adhesive composition is clear as measured by ASTM D 2090.
28. The moisture curable adhesive composition of claim 27 wherein:
- the filler has a surface area of less than about 90 m²/g.
29. The moisture curable adhesive composition of claim 28 wherein:
- the particle size of the filler ranges from about 40 nanometers to about 60 nanometers.
30. The moisture curable adhesive composition of claim 24 wherein the filler is fumed silica.
31. The moisture curable adhesive composition of claim 24 wherein:
- the moisture curable adhesive composition does not exhibit visible yellowing and cracking after about 1000 hours in the Q-UV Weatherometer.
32. The moisture curable adhesive composition of claim 30 further comprising:
- from about 0.01% to about 7% by weight of a second filler having a surface area that is at least about 90 m²/g.
33. The moisture curable adhesive composition of claim 24 wherein:
- the polymer is selected from the group consisting of: polyalkyl oxide, polyalkane, polyalkene, polyalkyne, substituted alkyl monomers, and mixtures thereof; and

the reactive silicone group is selected from the group consisting of: polyalkyl oxide, polyalkane, polyalkene, polyalkyne, substituted alkyl monomers, and mixtures thereof; and

the catalyst is selected from the group consisting of: stannous octoate, dibutyltin diacetate, dibutyltin dilaurate, dibutyltin mercaptide, dibutyltin thiocaboxylate, dibutyltin dimaleate, dioctyltin mercaptide, dioctyltin thiocarboxylate, dibutyl tin oxide in diisooctyl phthalate, aliphatic organotin, and mixtures thereof.

34. The moisture curable composition of claim 33 wherein the filler has a surface area that ranges from about 35 m²/g to about 65 m²/g.

35. The moisture curable composition of claim 34 wherein the filler is fumed silica.

36. A moisture curable adhesive composition comprising:

from about 50% to about 90% by weight polymer with reactive silicone end group, based on the total weight of the composition;

from about 0.1% to about 4% by weight catalyst, based on the total weight of the moisture curable composition;

from about 0.01% to about 35% by weight filler having a surface area less than about 90 meters squared per gram, based on the total weight of moisture curable composition;

from about 0.01% to about 5% by weight adhesion promoter, based on the total weight of the moisture curable composition; and

from about 0.01% to about 10% by weight dehydrating agent, based on the total weight of the moisture curable composition.

37. The moisture curable adhesive composition of 36 wherein:

the polymer is selected from the group consisting of: polyalkyl oxide, polyalkane, polyalkene, polyalkyne, substituted alkyl monomers, and mixtures thereof; and

the reactive silicone group is selected from the group consisting of: polyalkyl oxide, polyalkane, polyalkene, polyalkyne, substituted alkyl monomers, and mixtures thereof; and

the catalyst is selected from the group consisting of: stannous octoate, dibutyltin diacetate, dibutyltin dilaurate, dibutyltin mercaptide, dibutyltin thiocboxylate, dibutyltin dimaleate, dioctyltin mercaptide, dioctyltin thiocarboxylate, dibutyl tin oxide in diisooctyl phthalate, aliphatic organotin, and mixtures thereof.

38. The moisture curable adhesive composition of claim 37 wherein the filler is fumed silica having a surface area that is less than about 90 m²/g.

39. A method of joining a first adherend and a second adherend comprising the steps of:

applying a moisture curable adhesive comprising a polymer having a reactive silicon end group, a catalyst and a diluent, to at least one adherend;

maintaining the first adherend and the second adherend in non-contact with each other for less than about 20 minutes until the adhesive begins to cure; and

placing a second adherend in contact with the moisture curable adhesive applied to the first adherend to form a bond between the two adherends.

40. The method of claim 39 wherein:

at least one of the first adherend and the second adherend can be repositioned within five minutes of placing the second adherend on contact with the first adherend.