

Description

The present invention relates to silicone foam compositions. More particularly, the present invention relates to a method for reducing the density of silicone foam compositions.

Elastomeric silicone foams are well known in the art. Reference may be made, for example, to U.S. Patent Nos. 4,189,545; 3,923,705, 4,599,367 and 3,436,366. These foams have excellent physical properties and are useful in a variety of applications, for example, thermal and electrical insulation, flame barriers, and cushions.

However, elastomeric silicone foams have the drawback of being expensive. Thus, although these foams are superior to organic foams in many instances, organic foams are often used instead for economic reasons.

In an effort to overcome the cost disadvantages of silicone foams over organic foams, silicone foam manufacturers are constantly developing techniques for reducing the density of the silicone foam. Methods for reducing the density of silicone foam compositions are disclosed, for example, in U.S. Patent Nos. 4,418,157, 4,851,452 and 4,879,317.

U.S. Patent 4,879,317 to Smith et al. (Smith), discloses that a substantial reduction in silicone foam density can be achieved by using a hydride polysiloxane in a silicone foamable mixture comprising a vinyl-terminated polydiorganosiloxane, a hydride polysiloxane, a hydroxylated material, and an effective amount of a platinum catalyst, wherein the hydride polysiloxane in such silicone foamable mixture consists essentially of a mixture of a linear hydride polysiloxane and a cyclic hydride polysiloxane. A further reduction is achieved in the Smith foamable composition by using an aqueous mixture of a C_{11-8} alkanol, such as a mixture of water and methanol. It is stated in Smith that densities of less than 320.4 kg/m^3 (20 pounds per cubic feet) can be obtained by the method disclosed therein. Densities in the range of 120.1 to 225.9 kg/m^3 (7.5 to 14.1 pounds per cubic feet) are reported in the examples to have been obtained.

U.S. Patent No. 4,418,157 to Modic discloses a method for reducing the density of a silicone foam by mixing a foamable composition containing a polydiorganosiloxane base polymer, an organohydrogen siloxane, and a platinum catalyst, with a resinous copolymer selected from the group consisting of copolymers comprising $R_3\text{SiO}_{0.5}$ units and SiO_2 units, and copolymer comprising $R_3\text{SiO}_{0.5}$ units, $R_2^1\text{SiO}$ units and SiO_2 units. Densities of 299.5 and 411.7 kg/m^3 (18.7 and 25.7 pounds per cubic foot) were obtained in the examples in Modic.

U.S. Patent No. 4,851,452 to Gross et al. discloses a method for reducing the density of silicone foam by using a platinum catalyst in the form of a mixture of a platinum vinyl siloxane complex and a platinum triaryl phosphine to generate hydrogen in a foamable composition vinyl-terminated polydiorganosiloxane, a hydride polysiloxane and a hydroxylated material. Densities ranging from 179.4 to 280.3 kg/m^3 (11.2 to 17.5 pounds per cubic foot) were obtained in the examples.

It is desirable to provide a method for obtaining silicone foam having reduced density.

It is further desirable to provide a method for obtaining silicone foam having a density of 80 to 96 kg/m^3 (5 to 6 pounds per cubic foot).

Accordingly, it is an object of the present invention to provide a method for obtaining silicone foam having reduced density.

It is another object of the present invention to provide a method for obtaining silicone foam having a density of 80 to 96 kg/m^3 (5 to 6 pounds per cubic foot).

These objects are achieved in the present invention.

This invention is based on the discovery that the density obtained in a silicone foam can be reduced by controlling the pH of the foamable silicone composition used in preparing the foam.

The present invention provides a method for preparing a silicone foam having a reduced density of 80 to 96 kg/m^3 (5 to 6 pounds per cubic foot), comprising the step of agitating a mixture of ingredients comprising by weight:

(A) a silicone composition comprising by weight:

$$\begin{array}{c} \text{R}^1 \quad \text{R}^1 \quad \text{R}^1 \\ | \quad | \quad | \\ \text{R} - \text{Si} - \text{O} - \text{Si} - \text{O} - \text{Si} - \text{R} \\ | \quad | \quad | \\ \text{R}^1 \quad \text{R}^1 \quad \text{R}^1 \\ \quad \quad \quad \times \end{array}$$

(B) 2.5 to 20 parts of a substantially linear hydride polysiloxane having the formula

$$\begin{array}{c} \begin{array}{c} \text{R}^3 \\ | \\ \text{R}^2 - \text{Si} - \text{O} - \text{Si} - \text{O} - \text{Si} - \text{R}^2 \\ | \quad \quad | \quad \quad | \\ \text{R}^3 \quad \quad \text{H} \quad \quad \text{R}^3 \end{array} \\ \begin{array}{ccc} & \text{u} & \text{v} \end{array} \end{array}$$

(C) 0.1 to 15 parts of a cyclic hydride polysiloxane having the general formula:

$$\begin{array}{c} \text{R}^6 \\ | \\ \text{SiO} \\ | \\ \text{R}^6 \end{array} \quad y \quad \begin{array}{c} \text{R}^6 \\ | \\ \text{SiO} \\ | \\ \text{H} \end{array} \quad z$$

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of 0 to 5 inclusive, z is an integer having a value of from 1 to 8 inclusive, and the sum of y and z has a value of 3 to 8 inclusive.

(D) 0.1 to 5 parts of a hydroxyl source selected from the group consisting of water, organic alcohol, or a mixture of the foregoing.

The present invention is further directed to a foamable silicone composition comprising ingredients (A)-(D) used in the method set forth above as well as to the silicone foams resulting from the cure of the foamable compositions.

Part (1) of Component A contains 100 parts of a vinyl-terminated polydiorganosiloxane having formula (I) above.

In formula (I), R and R¹ may be substituted or unsubstituted hydrocarbon radicals of from 1 to 20 carbon atoms with the proviso that there is controlled vinyl substitution and controlled viscosity. The vinyl substitution should range from 0.0002 to 3% by weight and preferably, from about 0.001 to about 1% by weight.

Viscosity should vary from 100 to 1,000,000 centipoise at 25 °C. and preferably, from about 2500 to 500,000 centipoise. Although a polymer having a broader range of vinyl content will operate in the present invention, the more limited vinyl concentration given above will allow for a reaction that proceeds at an appropriate rate, that is not too slow and allows for proper cross-linking in the polymer to produce cured elastomeric silicone foam. With respect to the preferred range of viscosity, as can be appreciated, it is preferred that the viscosity not be too high, otherwise the composition is difficult to handle and will not foam well. The vinyl-containing polymer will form the major part of the foamable composition and thus the viscosity will be important to the viscosity of the final composition.

Substituted or unsubstituted hydrocarbon radicals of R and R¹ may be selected from alkyl radicals, methyl, ethyl, propyl, etc.; cycloalkyl radicals such as cyclohexyl, cycloheptyl, etc.; mononuclear aryl radicals such as phenyl, methyl phenyl, ethylphenyl, etc.; alkenyl radicals such as vinyl, allyl, etc. and more preferably vinyl and other well known substituent groups for diorganopolysiloxane polymers. Preferably the R and R¹ radicals are selected from the class consisting of alkyl radicals of 1 to 8 carbon atoms, aryl radicals such as phenyl; vinyl radicals and halogenated hydrocarbon atoms, such as, for instance 3,3,3-trifluoropropyl. Most phenyl radicals, vinyl radicals and 3,3,3-trifluoropropyl radicals where the polymer has the foregoing vinyl substitution and R is vinyl. Although vinyl-containing polysiloxane where the vinyl units appear only on the siloxane chain can be used to produce silicone foam, the physical properties are not as good as where there is terminal vinyl substitution. A particularly preferred polymer has only terminal vinyl substitution, i.e. where R is vinyl and R¹ is methyl, phenyl, and/or 3,3,3-trifluoropropyl.

Vinyl-containing polysiloxanes are well known in the art and may be produced by a variety of methods, for instance, by equilibrating cyclic polysiloxanes with or without vinyl substitution in the presence of appropriate amounts of vinyl substituted chainstopper. Further details for production of vinyl-substituted polysiloxanes may be found in U.S. Pat. No. 3,425,967 to Modic. Further suitable vinyl-containing polysiloxanes may be found in U.S. Pat. Nos. 4,599,367; 4,418,157 and 3,923,705.

Part (2) of Component A contains 1 to 5 parts of a buffer solution having a pH in the range of 9 to 10, and preferably about 10. The buffer solution is preferably present in the foamable composition in an amount ranging from 0.5 to 5, and most preferably 2 to 4, parts by weight.

It is to be understood by those skilled in the art that the concentration of buffer in the buffer solution should not be excessive because too high a buffer concentration may result in extremely rapid hydrogen gas evolution but no cure, eventually leading to the collapse of the rising foam under its own weight. What constitutes excessive buffer concentration for a particular buffer solution can be determined by simple experimentation by those skilled in the art.

The aqueous buffer solutions useful in the present invention include any of the standard aqueous buffer solutions known in the art for providing pH values in the range of 9 to 10. Suitable buffer solutions are disclosed in the CRC Handbook of Chemistry and Physics, 70th Edition, at page D-145.

For example, a buffer solution containing 50 milliliters of 0.025 molar borax and 4.6 milliliters of 0.1 molar HCl can be used to provide a pH of 9. A buffer solution containing 50 milliliters of 0.05 molar sodium bicarbonate and 10.7 milliliters of 0.1 molar NaOH will provide a pH of 10.

Part (3) of component A contains 25 to 140 parts per million of a platinum catalyst. Suitable platinum catalysts include, for example, reaction products of an olefin and chloroplatinic acid as described in Ashby, U.S. Patent 3,159,601, or the reaction product of platinic chloride and cyclopropane as described in Ashby, U.S. Patent 3,159,662. Further platinum complexes which can be used as the platinum catalyst are reaction products of chloroplatinic acid with up to 2 moles, per gram of platinum of a member selected from the class consisting of alcohols, ethers, aldehydes, and mixtures thereof, as shown in Lamoreaux, U.S. Patent 3,220,972. The preferred platinum catalyst is shown by Karstedt, U.S. Patent 3,775,452, which is formed by

reacting chloroplatinic acid with tetramethyldivinyldisiloxane in the presence of sodium bicarbonate in an ethanol solution. It has been found that effective results can be achieved if sufficient platinum catalyst is used in the silicone foamable composition to provide from 1 to 250 parts per million of platinum, preferably from 1 to 200 parts of platinum per million parts of mixture.

5 In preferred embodiments of the present invention, component A comprises 10 to 100 parts by weight of a resinous copolymer selected from the group consisting of copolymers comprising $R_3^7 \text{SiO}_{0.5}$ units and SiO_2 units, the ratio of $R_3^7 \text{SiO}_{0.5}$ units to SiO_2 units ranging from about 0.25 to about 0.8:1, and copolymers comprising $R_3^7 \text{SiO}_{0.5}$ units, $R_2^8 \text{SiO}$ units and SiO_2 units, where the ratio of monofunctional units to tetrafunctional units is from 0.25 to 0.8:1 and the ratio of difunctional units to tetrafunctional units is from 0 to 0.1:1, where R^7 and R^8 are selected from the group consisting of alkyl radicals, aryl radicals, aralkyl radicals, cycloalkyl radicals, vinyl radicals, allyl radicals and fluoroalkyl radicals. The presence of this resinous copolymer acts to lower the density and increase the strength of the foam. This copolymer is known in the art, for example, as described in U.S. Patent No. 4,418,157 to Modic. Preferably, in the formulas provided above for the copolymer, the alkyl radicals have from 1 to 8 carbon atoms, the aralkyl radicals from 7 to 13 carbon atoms, the cycloalkyl radicals from 6 to 8 carbon atoms and preferably the fluoroalkyl radical is 3,3,3-trifluoropropyl.

Preferably, Component A also contains from 1 to 200 parts by weight of a filler per hundred parts of foam composition. It is preferred to use extending fillers or reinforcing fillers such as fumed silica. Precipitated silica also can be used in instances where it is desired to increase the physical properties such as the tensile strength and tear strength of the resulting elastomeric foam. Other extending fillers which may be utilized are, for example, titanium dioxide, lithopone, zinc oxide, zirconium silicate, silica airogel, iron oxide, diatomaceous earth, calcium carbonate, glass fibers, magnesium oxide, chromic oxide, zirconium oxide, aluminum oxide, alpha-quartz, clay, carbon and graphite. In order to minimize the viscosity increase generally experienced when using reinforcing fillers, the reinforcing fillers can be heat treated with cyclic polysiloxanes or silazanes. A preferred filler in the practice of this invention is ground quartz which has been found to enhance the burn-resistant properties of the composition as well as imparting some enhanced physical properties to the final cured silicone foam.

Component B is a linear hydride polysiloxane having the general formula (II) above. Preferably, the hydride polysiloxane consists essentially of chemically bonded organosiloxy units having silicon bonded hydrogen atoms attached to silicon to form the polysiloxane chain backbone.

The composition of this invention preferably further contains 0.1 to 15 parts of a cyclic hydride polysiloxane having the general formula (III) above. Preferably, the cyclic hydride polysiloxane is a hydride cyclotetrasiloxane.

In formulas (II) and (III), R^2 may be independently hydrogen, an alkyl radical of from 1 to 8 carbon atoms, an aryl radical from 1 to 8 carbon atoms or a haloalkyl radical of from 3 to 8 carbon atoms.

It is preferred that the hydride polysiloxanes have a hydrogen content ranging from about 0.3 to about 1.6% by weight and as shown in formula (II), u and v vary to provide a viscosity ranging from 1 to 500 centipoise at 25°C. The hydride content is not critical but such a content substantially less than about 0.3% by weight of the hydride polysiloxanes may not provide sufficient hydrogen in the foamable composition to react with the hydroxy source and liberate hydrogen gas as well as react and cross-link with vinyl molecules of the vinyl-containing polysiloxane. The result will of course be a poorly blown and undercured foam. Likewise, viscosity of the hydride polysiloxane is not critical, but higher viscosity materials may be difficult to obtain and utilize. Preferably, viscosity ranges from about 5 to about 250 centipoise at 25°C, and most preferably from about 5 to 100 centipoise.

Component D provides a hydroxyl source. A hydroxyl source is necessary to properly blow the foamable composition. The source of hydroxyl may be water or organic alcohol or a mixture of water and organic alcohol. Suitable hydroxylated materials include organic alcohols which are monohydric or polyhydric alcohols having from about 1-12 carbon atoms. Preferably, there can be used methanol, ethanol, propanol, butanol, lauryl alcohol, octyl alcohol, and ethylene glycol. The most preferred alcohol is methanol.

Other aspects of the present invention provide a foamable composition comprising components A-D above and the silicone foam produced from the cure thereof.

The silicone foamable compositions of the present invention are generally used and stored as a four-part composition, with components A, B, C, and D forming the four parts.

In forming the silicone foam, components B, C, and D are agitated, preferably rapidly, with component A. The resulting composition can be poured into a cavity and allowed to stand. The composition thereafter will begin to cross-link and generate gas simultaneously resulting in a cured elastomeric silicone foam having a density of 80 to 96 kg/m³ (5 to 6 pounds per cubic foot).

In order that those skilled in the art will be better able to practice the present invention, the following examples are given by way of illustration and not by way of limitation. All parts are by weight.

Examples 1-7 and Control Example A

A mixture of 200 grams of a blend (Part A) was prepared containing 39% by weight of a polydimethylsiloxane having terminal dimethylvinylsiloxyl units and a viscosity of about 75,000 centistokes, 15% by weight of a polydimethylsiloxane having terminal dimethylvinylsiloxyl units and a viscosity of 4000 centistokes, 18% by weight of a methylsiloxane resin consisting essentially of chemically combined SiO_2 in a ratio of 1.43 moles of trimethylsiloxyl units, per 0.25 mole of methylvinylsiloxyl units, 1.5% by weight of buffered water, and 20 to 45 parts per million of platinum in the form of a platinum vinyl siloxane complex. Example 7 further contained 25% by weight of alpha-quartz.

There was then added to Part A, 20.0 grams of a linear hydride polysiloxane having terminal trimethylsiloxyl units and consisting essentially of methyl hydrogen siloxyl units having a viscosity of about 15-40 centistokes (Part B), and 7.58 grams of a cyclic methylhydrogen tetrasiloxane (Part C), and 3.02 grams of methanol (Part D).

Because it has reduced polymer content as a result of filler loading as compared to Part A in Examples 1-6, Part A in Example 7 was converted to silicone foam by reacting it with 5.68 grams of a cyclic methylhydrogen tetrasiloxane, 15.0 grams of a linear hydride polysiloxane having terminal trimethylsiloxyl units and consisting essentially of methylhydrogen siloxyl units having a viscosity of about 15-40 centistokes, and 2.26 grams of methanol.

The hydride polysiloxane mixture was mixed with the platinum containing silicone mixture for 30 seconds and then transferred to a pre-weighed container. Foaming occurred in the resulting mixed blend.

The buffering solutions used in the respective examples are indicated in Table 1 below.

TABLE 1

Example No.	Description	pH of buffer solution
1	potassium hydrogen phthalate	5
2	potassium phosphate mono basic/sodium hydroxide	6
3	potassium phosphate mono basic/sodium hydroxide	7
4	potassium phosphate mono basic/sodium hydroxide	8
5	borax	9
6	sodium bicarbonate/sodium carbonate	10
7	sodium bicarbonate/sodium carbonate plus 335g of 10M minusil	10
A	deionized water -not buffered	-

The density of the foams prepared in Examples 1-7 and Control Example A were measured and are presented in Table 2 below.

TABLE 2

Example No.	pH of Buffer Solution	Density Kg/m ³ (lbs/ft ³)
1	5	115.3 (7.2)
2	6	107.3 (6.7)
3	7	112.1 (7.0)
4	8	89.7 (5.6)
5	9	96.1 (6.0)
6	10	76.9 (4.8)
7	10	76.9 (4.8)
A	-	147.3 (9.2)

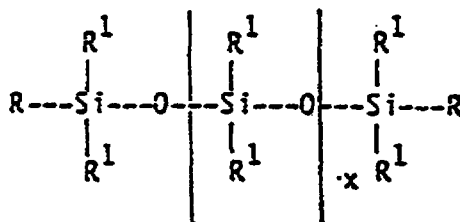
The data shown in Table 2 indicates that a silicone foam having a density of 80 to 96 kg/m³ (5 to 6 lbs/ft³) can be obtained at pH levels of 9 to 10. The data further shows that the presence of a filler does not affect the buffer solution's effects on the density.

5 Claims

1. A method for preparing a silicone foam having a reduced density of 80 to 96 kg/m³ (5 to 6 pounds per cubic foot), comprising the step of agitating a mixture of ingredients comprising by weight:

(A) a silicone composition, comprising by weight:

- (1) 100 parts of a vinyl-terminated polydiorganosiloxane of the formula:

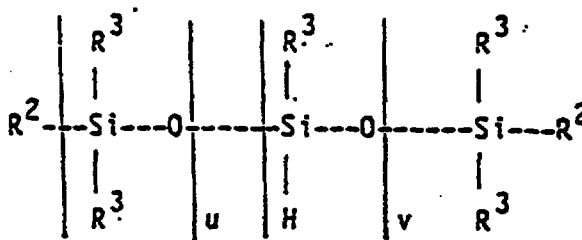


wherein R and R¹ are selected from the group consisting of substituted or unsubstituted hydrocarbon radicals or from 1 to 20 carbon atoms, such that the polymer contains from 0.0002 to 3% by weight vinyl and x varies such that the viscosity of the polymer varies from 100 to 1,000,000 centipoise at 25 °C;

- (2) 0.5 to 5 parts by weight of an aqueous buffer solution having a pH in the range of 9 to 10; and

(3) 25 to 140 parts per million of a platinum catalyst;

- (B) 2.5 to 20 parts of a substantially linear hydride polysiloxane having the formula



wherein R² is selected independently from the group consisting of hydrogen, alkyl radicals of from 1 to 8 carbon atoms, halo substituted alkyl radicals of from 1 to 8 carbon atoms, aryl radicals of from 6 to 14 carbon atoms, and halo substituted aryl radicals of from 6 to 14 carbon atoms; and R³ is selected from the group consisting of alkyl radicals of from 1 to 8 carbon atoms, aryl radicals of from 6 to 14 carbon atoms, halo aryl radicals of from 6 to 14 carbon atoms, and haloalkyl radicals of from 3 to 8 carbon atoms; u and v are integers which can vary sufficiently to provide a hydride polysiloxane having a viscosity of from 5 to 10,000 centipoise at 25 °C;



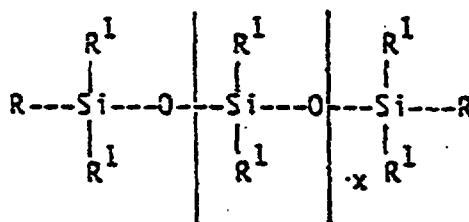
The data shown in Table 2 indicates that a silicone foam having a density of 80 to 96 kg/m³ (5 to 6 lbs/ft³) can be obtained at pH levels of 9 to 10. The data further shows that the presence of a filler does not affect the buffer solution's effects on the density.

5 Claims

1. A method for preparing a silicone foam having a reduced density of 80 to 96 kg/m³ (5 to 6 pounds per cubic foot), comprising the step of agitating a mixture of ingredients comprising by weight:

(A) a silicone composition, comprising by weight:

- (1) 100 parts of a vinyl-terminated polydiorganosiloxane of the formula:

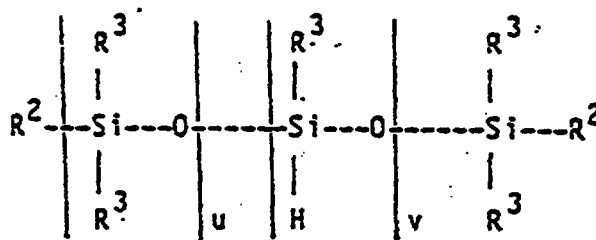


wherein R and R¹ are selected from the group consisting of substituted or unsubstituted hydrocarbon radicals or from 1 to 20 carbon atoms, such that the polymer contains from 0.0002 to 3% by weight vinyl and x varies such that the viscosity of the polymer varies from 100 to 1,000,000 centipoise at 25°C;

- (2) 0.5 to 5 parts by weight of an aqueous buffer solution having a pH in the range of 9 to 10; and

- (3) 25 to 140 parts per million of a platinum catalyst;

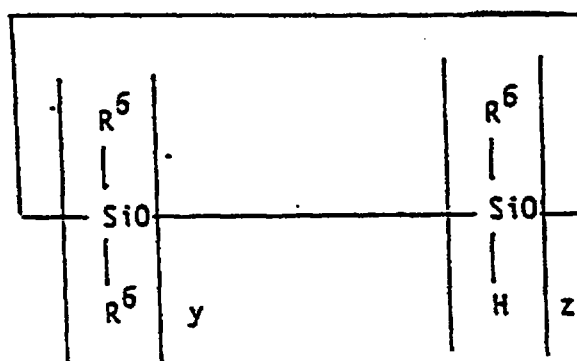
(B) 2.5 to 20 parts of a substantially linear hydride polysiloxane having the formula



wherein R² is selected independently from the group consisting of hydrogen, alkyl radicals of from 1 to 8 carbon atoms, halo substituted alkyl radicals of from 1 to 8 carbon atoms, aryl radicals of from 6 to 14 carbon atoms, and halo substituted aryl radicals of from 6 to 14 carbon atoms; and R³ is selected from the group consisting of alkyl radicals of from 1 to 8 carbon atoms, aryl radicals of from 6 to 14 carbon atoms, halo aryl radicals of from 6 to 14 carbon atoms, and haloalkyl radicals of from 3 to 8 carbon atoms; u and v are integers which can vary sufficiently to provide a hydride polysiloxane having a viscosity of from 5 to 10,000 centipoise at 25°C;



(C) 0.1 to 15 parts of a cyclic hydride polysiloxane having the general formula:



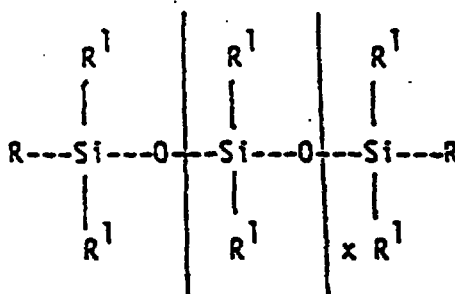
wherein R^5 may be independently hydrogen, an alkyl radical of from 1 to 8 carbon atoms, an aryl radical from 6 to 14 carbon atoms, or a haloalkyl radical of 3 to 8 carbon atoms; y is an integer having a value of 0 to 5 inclusive, z is an integer having a value of from 1 to 8 inclusive, and the sum of y and z has a value of 3 to 8 inclusive; and

(D) 0.1 to 5 parts of a hydroxyl source selected from the group consisting of water, organic alcohol, or a mixture of the foregoing.

2. The method of claim 1 wherein the pH of the mixture is 9 to 10.
3. The method of claim 2 wherein the pH of the mixture is about 10.
4. A method according to claim 1 wherein the aqueous buffer solution is added in the amount of 2 to 4 parts by weight.
5. The method of claim 1 wherein component A further comprises 10 to 100 parts by weight of a resinous copolymer selected from the group consisting of copolymers comprising $R_3^5 \text{SiO}_{0.5}$ units and SiO_2 units, the ratio of $R_3^5 \text{SiO}_{0.5}$ units to SiO_2 units ranging from 0.25 to 0.8:1, and copolymers comprising $R_3^5 \text{SiO}_{0.5}$ units, $R_2^5 \text{SiO}$ units and SiO_2 units, where the ratio of monofunctional units to tetrafunctional units is from 0.25 to 0.8:1 and the ratio of difunctional units to tetrafunctional units is from 0 to 0.1:1, where R^5 and R^6 are selected from the group consisting of alkyl radicals, aryl radicals, aralkyl radicals, cycloalkyl radicals, vinyl radicals, allyl radicals and fluoroalkyl radicals.
6. A method according to claim 1 wherein the silicone foam composition further comprises 1 to 200 parts by weight of a reinforcing or extending filler per hundred parts of foamable composition.
7. A method according to claim 6 wherein the filler is alpha-ground quartz.
8. The method of claim 1 wherein the vinyl terminated polydiorganosiloxane is a vinyl terminated polydimethylsiloxane.
9. The method of claim 1 wherein the hydroxyl source is an alkanol having 1 to 8 carbon atoms.
10. The method of claim 9 wherein the alkanol is methanol.
11. The method of claim 1 wherein the cyclic hydride polysiloxane is a hydride cyclotetrasiloxane.
12. The method of claim 1 wherein the platinum catalyst is a platinum vinylidisiloxane.
13. A foamable composition comprising by weight:
 - (A) a silicone composition, comprising by weight:



(1) 100 parts of a vinyl-terminated polydiorganosiloxane of the formula:

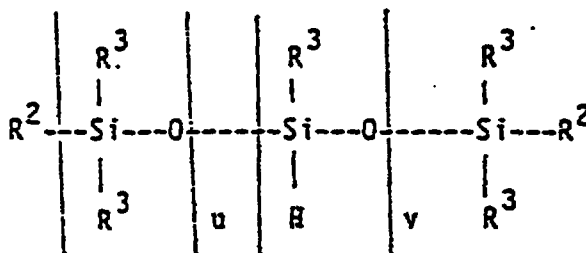


wherein R and R¹ are selected from the group consisting of substituted or unsubstituted hydrocarbon radicals of from 1 to 20 carbon atoms, such that the polymer contains from 0.0002 to 3% by weight vinyl and x varies such that the viscosity of the polymer varies from 100 to 1,000,000 centipoise at 25°C;

(2) 0.5 to 5 parts by weight of an aqueous buffer solution having a pH in the range of 9 to 10; and

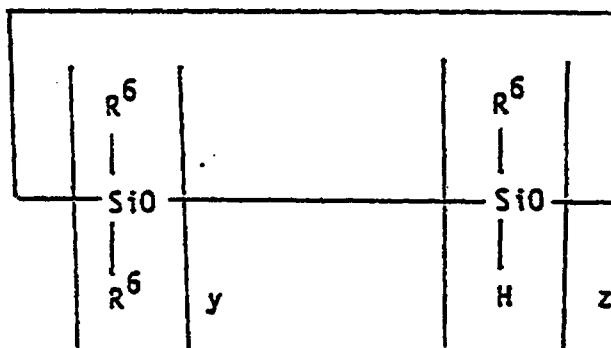
(3) 25 to 140 parts per million of a platinum catalyst;

(B) 2.5 to 20 parts of a substantially linear hydride polysiloxane having the formula



wherein R² is selected independently from the group consisting of hydrogen, alkyl radicals of from 1 to 8 carbon atoms, halo substituted alkyl radicals of from 1 to 8 carbon atoms, aryl radicals of from 6 to 14 carbon atoms, and halo substituted aryl radicals of from 6 to 14 carbon atoms; and R³ is selected from the group consisting of alkyl radicals of from 1 to 8 carbon atoms, aryl radicals of from 6 to 14 carbon atoms, halo aryl radicals of from 6 to 14 carbon atoms, and haloalkyl radicals of from 3 to 8 carbon atoms; u and v are integers which can vary sufficiently to provide a hydride polysiloxane having a viscosity of from 5 to 10,000 centipoise at 25°C;

(C) 0.1 to 15 parts of a cyclic hydride polysiloxane having the general formula:



wherein R⁶ may be independently hydrogen, an alkyl radical of from 1 to 8 carbon atoms, an aryl radical from 6 to 14 carbon atoms, or a haloalkyl radical of 3 to 8 carbon atoms; y is an integer having a value of 0 to 5 inclusive, z is an integer having a value of from 1 to 8 inclusive, and the sum of y and z has a value of 3 to 8 inclusive; and



(D) 0.1 to 5 parts of a hydroxyl source selected from the group consisting of water, organic alcohol, or a mixture of the foregoing.

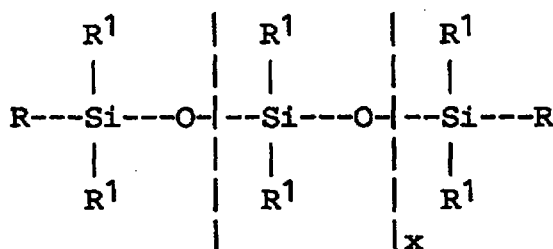
14. A silicone foam having a reduced density of 80 to 96 kg/m³ (5 to 6 pounds per cubic foot), comprising the cured product of claim 13.

Patentansprüche

1. Verfahren zum Herstellen eines Siliconschaumes mit verringerter Dichte von 80 bis 96 kg/m³ (5-6 pounds per cubic foot), umfassend die Stufe des Rührens einer Mischung von Bestandteilen, umfassend, bezogen auf das Gewicht:

(A) eine Silicon-Zusammensetzung, umfassend, bezogen auf das Gewicht:

(1) 100 Teile eines Vinyl-Endgruppen aufweisenden Polydiorganosiloxans der Formel:

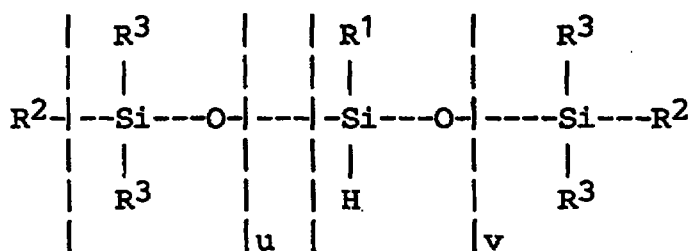


worin R und R¹ ausgewählt sind aus der Gruppe bestehend aus substituierten oder unsubstituierten Kohlenwasserstoffresten von 1 bis 20 Kohlenstoffatomen, derart, daß das Polymer von 0,0002 bis 3 Gew.-% Vinyl enthält und x derart variiert, daß die Viskosität des Polymers von 100 bis 1.000.000 mPa·s (centipoise) bei 25 °C variiert;

(2) 0,5 bis 5 Gewichtsteile einer wässrigen Pufferlösung mit einem pH im Bereich von 9 bis 10 und

(3) 25 bis 140 ppm eines Platin-Katalysators;

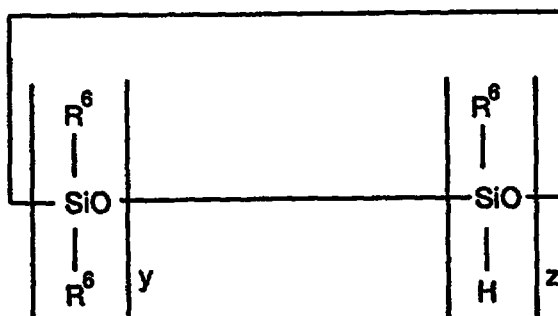
(B) 2,5 bis 20 Teile eines im wesentlichen linearen Hydridpolysiloxans der Formel



worin R² unabhängig ausgewählt ist aus der Gruppe bestehend aus Wasserstoff, Alkylresten von 1 bis 8 Kohlenstoffatomen, Halogen-substituierten Alkylresten von 1 bis 8 Kohlenstoffatomen, Arylresten von 6 bis 14 Kohlenstoffatomen und Halogen-substituierten Arylresten von 6 bis 14 Kohlenstoffatomen, und R³ ausgewählt ist aus der Gruppe bestehend aus Alkylresten mit 1 bis 8 Kohlenstoffatomen, Arylresten mit 6 bis 14 Kohlenstoffatomen, Halogenarylresten mit 6 bis 14 Kohlenstoffatomen und Halogenalkylresten mit 3 bis 8 Kohlenstoffatomen; u und v ganze Zahlen sind, die in genügender Weise variieren, um ein Hydridpolysiloxan mit einer Viskosität von 5 bis 10.000 mPa·s (centipoise) bei 25 °C zu schaffen;



(C) 0,1 bis 15 Teile eines cyclischen Hydridpolysiloxans der allgemeinen Formel:

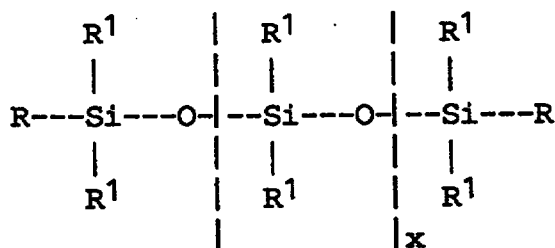


worin R^6 unabhängig Wasserstoff, ein Alkylrest von 1 bis 8 Kohlenstoffatomen, ein Arylrest von 6 bis 14 Kohlenstoffatomen oder ein Halogenalkylrest von 3 bis 8 Kohlenstoffatomen sein kann; y eine ganze Zahl mit einem Wert von 0 bis einschließlich 5 ist, z eine ganze Zahl mit einem Wert von 1 bis einschließlich 8 ist, und die Summe von y und z einen Wert von 3 bis einschließlich 8 hat und
(D) 0,1 bis 5 Teile einer Hydroxylquelle, ausgewählt aus der Gruppe bestehend aus Wasser, organischem Alkohol oder einer Mischung der vorgenannten.

2. Verfahren nach Anspruch 1, worin der pH der Mischung 9 bis 10 beträgt.
3. Verfahren nach Anspruch 2, worin der pH der Mischung etwa 10 beträgt.
4. Verfahren nach Anspruch 1, worin die wässrige Pufferlösung in der Menge von 2 bis 4 Gewichtsteilen hinzugegeben wird.
5. Verfahren nach Anspruch 1, worin Komponente A weiter 10 bis 100 Gewichtsteile eines harzartigen Copolymers umfaßt, ausgewählt aus der Gruppe bestehend aus Copolymeren, umfassend $\text{R}^5_3\text{SiO}_{0,5}$ -Einheiten und SiO_2 -Einheiten, das Verhältnis der $\text{R}^5_3\text{SiO}_{0,5}$ -Einheiten zu den SiO_2 -Einheiten im Bereich von 0,25 bis 0,8:1 liegt und Copolymeren, umfassend $\text{R}^5_3\text{SiO}_{0,5}$ -Einheiten, R^6_2SiO -Einheiten und SiO_2 -Einheiten, worin das Verhältnis der monofunktionellen Einheiten zu den tetrafunktionellen Einheiten im Bereich von 0,25 bis 0,8:1 liegt und das Verhältnis der difunktionellen Einheiten zu den tetrafunktionellen Einheiten im Bereich von 0 bis 0,1:1 liegt, worin R^5 und R^6 ausgewählt sind aus der Gruppe bestehend aus Alkylresten, Arylresten, Aralkylresten, Cycloalkylresten, Vinylresten, Allylresten und Fluoralkylresten.
6. Verfahren nach Anspruch 1, worin die Siliconschaum-Zusammensetzung weiter 1 bis 200 Gewichtsteile eines verstärkenden oder streckenden Füllstoffes auf 100 Teile schäumbare Zusammensetzung umfaßt.
7. Verfahren nach Anspruch 6, worin der Füllstoff gemahlener α -Quarz ist.
8. Verfahren nach Anspruch 1, worin das Vinyl-Endgruppen aufweisende Polydiorganosiloxan ein Vinyl-Endgruppen aufweisendes Polydimethylsiloxan ist.
9. Verfahren nach Anspruch 1, worin die Hydroxylquelle ein Alkanol mit 1 bis 8 Kohlenstoffatomen ist.
10. Verfahren nach Anspruch 9, worin der Alkanol Methanol ist.
11. Verfahren nach Anspruch 1, worin das cyclische Hydridpolysiloxan Hydridcyclotetrasiloxan ist.
12. Verfahren nach Anspruch 1, worin der Platinkatalysator ein Platinvinylsiloxan ist.
13. Schäumbare Zusammensetzung, umfassend, bezogen auf das Gewicht:
 - (A) eine Silicon-Zusammensetzung, umfassend, bezogen auf das Gewicht:



(1) 100 Teile eines Vinyl-Endgruppen aufweisenden Polydioganosiloxans der Formel:

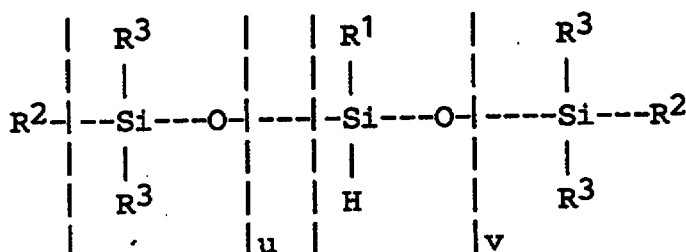


worin R und R¹ ausgewählt sind aus der Gruppe bestehend aus substituierten oder unsubstituierten Kohlenwasserstoffresten von 1 bis 20 Kohlenstoffatomen, derart, daß das Polymer von 0,0002 bis 3 Gew.-% Vinyl enthält und x derart variiert, daß die Viskosität des Polymers von 100 bis 1.000.000 mPa·s (centipoise) bei 25 °C variiert;

(2) 0,5 bis 5 Gewichtsteile einer wässrigen Pufferlösung mit einem pH im Bereich von 9 bis 10 und

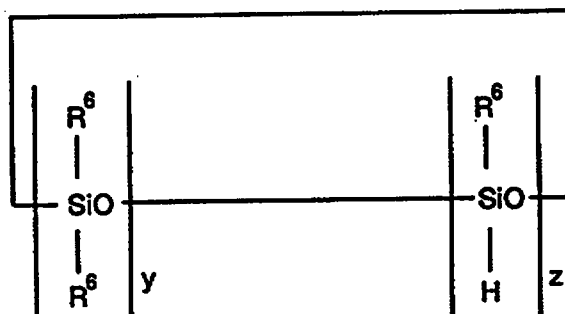
(3) 25 bis 140 ppm eines Platin-Katalysators;

(B) 2,5 bis 20 Teile eines im wesentlichen linearen Hydridpolysiloxans der Formel



worin R² unabhängig ausgewählt ist aus der Gruppe bestehend aus Wasserstoff, Alkylresten von 1 bis 8 Kohlenstoffatomen, Halogen-substituierten Alkylresten von 1 bis 8 Kohlenstoffatomen, Arylresten von 6 bis 14 Kohlenstoffatomen und Halogen-substituierten Arylresten von 6 bis 14 Kohlenstoffatomen, und R³ ausgewählt ist aus der Gruppe bestehend aus Alkylresten mit 1 bis 8 Kohlenstoffatomen, Arylresten mit 6 bis 14 Kohlenstoffatomen, Halogenarylresten mit 6 bis 14 Kohlenstoffatomen und Halogenalkylresten mit 3 bis 8 Kohlenstoffatomen; u und v ganze Zahlen sind, die in genügender Weise variieren, um ein Hydridpolysiloxan mit einer Viskosität von 5 bis 10.000 mPa·s (centipoise) bei 25 °C zu schaffen;

(C) 0,1 bis 15 Teile eines cyclischen Hydridpolysiloxans der allgemeinen Formel:



worin R⁶ unabhängig Wasserstoff, ein Alkylrest von 1 bis 8 Kohlenstoffatomen, ein Arylrest von 6 bis 14 Kohlenstoffatomen oder ein Halogenalkylrest von 3 bis 8 Kohlenstoffatomen sein kann; y eine ganze Zahl mit einem Wert von 0 bis einschließlich 5 ist, z eine ganze Zahl mit einem Wert von 1 bis einschließlich 8 ist, und die Summe von y und z einen Wert von 3 bis einschließlich 8 hat und



(D) 0,1 bis 5 Teile einer Hydroxylquelle, ausgewählt aus der Gruppe bestehend aus Wasser, organischem Alkohol oder einer Mischung der vorgenannten.

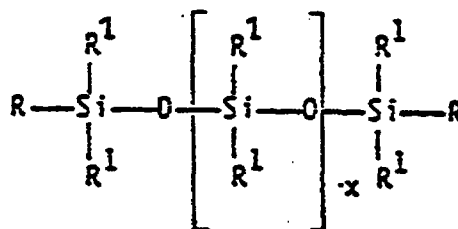
14. Siliconschaum mit verringerter Dichte von 80 bis 96 kg/m³ (5-6 pounds per cubic foot), umfassend das gehärtete Produkt von Anspruch 13.

Revendications

1. Procédé de préparation d'une mousse de silicone ayant une masse volumique réduite comprise entre 80 et 96 kg/m³ (5 à 6 livres par pied³), comprenant l'étape consistant à agiter un mélange d'ingrédients comprenant, en poids :

(A) une composition de silicone contenant, en poids

- (1) 100 parties d'un polydiorganosiloxane à terminaison vinyle correspondant à la formule

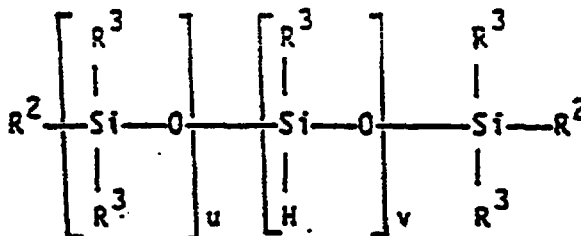


dans laquelle R et R¹ sont choisis dans le groupe formé par les radicaux hydrocarbonés substitués ou non comportant de 1 à 20 atomes de carbone, de manière à ce que le polymère contienne entre 0,0002 et 3 % en poids de groupes vinyle et x varie de manière telle que la viscosité du polymère varie entre 100 et 1 000 000 centipoises à 25 °C,

- (2) de 0,5 à 5 parties en poids d'une solution tampon aqueuse ayant un pH compris entre 9 et 10, et

(3) de 25 à 140 parties par million d'un catalyseur à base de platine,

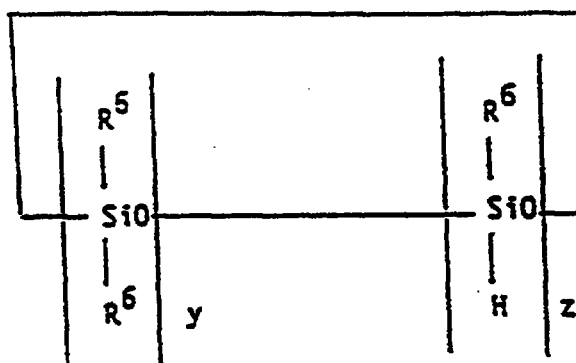
- (B) de 2,5 à 20 parties d'un hydrogénéo-polysiloxane pratiquement linéaire de formule :



dans laquelle R² est choisi indépendamment dans le groupe formé par un atome d'hydrogène, les radicaux alkyle comportant de 1 à 8 atomes de carbone, les radicaux alkyle halogénés comportant de 1 à 8 atomes de carbone, les radicaux aryle comportant de 6 à 14 atomes de carbone et les radicaux aryle halogénés comportant de 6 à 14 atomes de carbone, et R³ est choisi dans le groupe formé par les radicaux alkyle comportant de 1 à 8 atomes de carbone, les radicaux aryle comportant de 6 à 14 atomes de carbone, les radicaux aryle halogénés comportant de 6 à 14 atomes de carbone et les radicaux alkyle halogénés comportant de 3 à 8 atomes de carbone, u et v sont des nombres entiers qui peuvent varier suffisamment pour que l'hydrogénéo-polysiloxane ait une viscosité comprise entre 5 et 10 000 centipoises à 25 °C.



(C) de 0,1 à 15 parties d'un hydrogéo-polysiloxane cyclique répondant à la formule générale :



dans laquelle R^5 représente indépendamment un atome d'hydrogène, un radical alkyle comportant de 1 à 8 atomes de carbone, un radical aryle comportant de 6 à 14 atomes de carbone ou un radical halogénoalkyle comportant de 3 à 8 atomes de carbone, y est un nombre entier valant entre 0 et 5 inclus, z est un nombre entier valant entre 1 et 8 inclus, et la somme de y et z a une valeur comprise entre 3 et 8 inclus, et

(D) de 0,1 à 5 parties d'une source de fonctions hydroxyle choisie dans le groupe formé par l'eau, les alcools organiques ou un mélange des précédents.

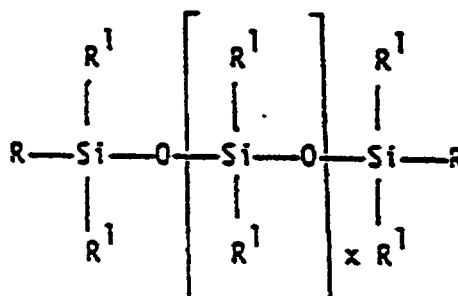
2. Procédé conforme à la revendication 1 dans lequel le pH du mélange est compris entre 9 et 10.
3. Procédé conforme à la revendication 2 dans lequel le pH du mélange est d'environ 10.
4. Procédé conforme à la revendication 1 dans lequel la solution tampon aqueuse est ajoutée à raison de 2 à 4 parties en poids.
5. Procédé conforme à la revendication 1 dans lequel le composant (A) comprend en outre de 10 à 100 parties en poids d'un copolymère résineux choisi dans le groupe formé par les copolymères contenant des motifs $R^5_3SiO_{0,5}$ et des motifs SiO_2 , le rapport des motifs $R^5_3SiO_{0,5}$ aux motifs SiO_2 étant compris entre 0,25/1 et 0,8/1, et les copolymères contenant des motifs $R^5_3SiO_{0,5}$ et des motifs R^6_2SiO , le rapport des motifs monofonctionnels aux motifs tétrafonctionnels étant compris entre 0,25/1 et 0,8/1, et le rapport des motifs difonctionnels aux motifs tétrafonctionnels étant compris entre 0 et 0,1/1, R^5 et R^6 étant choisis dans le groupe formé par les radicaux alkyle, aryle, aralkyle, cycloalkyle, vinyle, allyle et fluoroalkyle.
6. Procédé conforme à la revendication 1 dans lequel la composition de mousse de silicone contient en outre de 1 à 200 parties en poids d'une charge renforçante ou diluante pour 100 parties en poids de composition expansible.
7. Procédé conforme à la revendication 6 dans lequel la charge est du quartz alpha broyé.
8. Procédé conforme à la revendication 1 dans lequel le polydiorganosiloxane à groupes vinyle terminaux est un polydiméthylsiloxane à groupes vinyle terminaux.
9. Procédé conforme à la revendication 1 dans lequel la source de fonctions hydroxyle est un alcool comportant de 1 à 8 atomes de carbone.
10. Procédé conforme à la revendication 9 dans lequel l'alcool est le méthanol.
11. Procédé conforme à la revendication 1 dans lequel l'hydrogéo-polysiloxane cyclique est un hydrogéo-tétrasiloxane cyclique.
12. Procédé conforme à la revendication 1 dans lequel le catalyseur de platine est un vinylsiloxane de platine.



13. Composition expansible comprenant, en poids :

(A) une composition silicone contenant, en poids

(1) 100 parties d'un polydiorganosiloxane à terminaisons vinyle correspondant à la formule

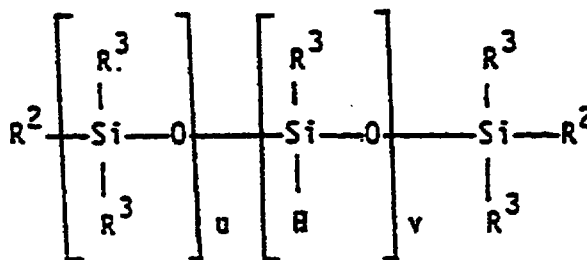


dans laquelle R et R¹ sont choisis dans le groupe formé par les radicaux hydrocarbonés substitués ou non comportant de 1 à 20 atomes de carbone, de manière à ce que le polymère contienne entre 0,0002 et 3% en poids de groupes vinyle et x varie de manière telle que la viscosité du polymère varie entre 100 et 1 000 000 centipoises à 25 °C,

(2) de 0,5 à 5 parties en poids d'une solution tampon aqueuse ayant un pH compris entre 9 et 10, et

(3) de 25 à 140 parties par million d'un catalyseur à base de platine,

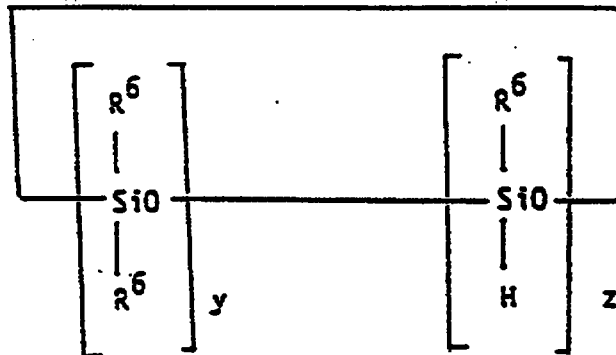
(B) de 2,5 à 20 parties d'un hydrogénéopolysiloxane pratiquement linéaire de formule :



dans laquelle R² est choisi indépendamment dans le groupe formé par un atome d'hydrogène, les radicaux alkyle comportant de 1 à 8 atomes de carbone, les radicaux alkyle halogénés comportant de 1 à 8 atomes de carbone, les radicaux aryle comportant de 6 à 14 atomes de carbone et les radicaux aryle halogénés comportant de 6 à 14 atomes de carbone, et R³ est choisi dans le groupe formé par les radicaux alkyle comportant de 1 à 8 atomes de carbone, les radicaux aryle comportant de 6 à 14 atomes de carbone, les radicaux aryle halogénés comportant de 6 à 14 atomes de carbone et les radicaux alkyle halogénés comportant de 3 à 8 atomes de carbone, u et v sont des nombres entiers qui peuvent varier suffisamment pour que l'hydrogénéopolysiloxane ait une viscosité comprise entre 5 et 10 000 centipoises à 25 °C.



(C) de 0,1 à 15 parties d'un hydrogéo-polysiloxane cyclique répondant à la formule générale :



dans laquelle R^6 représente indépendamment un atome d'hydrogène, un radical alkyle comportant de 1 à 8 atomes de carbone, un radical aryle comportant de 6 à 14 atomes de carbone ou un radical halogénoalkyle comportant de 3 à 8 atomes de carbone, y est un nombre entier valant entre 0 et 5 inclus, z est un nombre entier valant entre 1 et 8 inclus, et la somme de y et z a une valeur comprise entre 3 et 8 inclus, et

(D) de 0,1 à 5 parties d'une source de fonctions hydroxyle choisie dans le groupe formé par l'eau, les alcools organiques ou un mélange des précédents.

14. Mousse de silicone ayant une masse volumique réduite comprise entre 80 et 96 kg/m³ (5 à 6 livres par pied³) contenant le produit durci défini par la revendication 13.