Title: HYDROXYLAMINE-TERMINATED POLYSILOXANE AND METHODS OF MAKING AND USING THE SAME

Abstract: The present invention relates to hydroxylamine-terminated polysiloxanes. In various embodiments, the present invention provides a hydroxylamine-terminated polysiloxane having no pendant amine-functional groups. The present invention also provides methods of making the polysiloxane, compositions and articles of manufacture including the polysiloxane, and methods of using the polysiloxane.
HYDROXYLAMINE-TERMINATED POLYSILOXANE AND METHODS OF MAKING AND USING THE SAME

CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application claims the benefit of priority to U.S. Provisional Patent Application Serial No. 61/759,452 filed Feb. 1, 2013, the disclosure of which is incorporated herein in its entirety by reference.

[0002] Silicones have been used extensively for fiber or textile treatments. Amine-functional groups have favorable interactions between amine-functional groups and fibers or textiles. However, disadvantages of amine-functionalized polysiloxanes can include yellowing due to oxidation of the amine groups. Techniques have been used to incorporate pendant (e.g. non-terminal) amine functionality into polysiloxanes, such as equilibration techniques. However, ensuring complete equilibration (e.g. randomization of amine-functionality) can require long periods of time, and it can be difficult to assess whether the amine-functionality has become sufficiently randomized in the polymer. In addition, some polysiloxane chains may not receive any amine functionality during the equilibration.

SUMMARY OF THE INVENTION

[0003] In various embodiments, the present invention provides a hydroxylamine-terminated polysiloxane having the structure \( E-R^3-\left[\text{SiR}^1\text{R}^2\text{O}\right]_{DP}\text{SiR}^1\text{R}^2-R^3-E \). The variable \( E \) is a monovalent hydroxylamino functional group. Each of \( R^1 \) and \( R^2 \) is at each occurrence independently at least one of straight chain, branched, and cyclic, substituted or unsubstituted (C\(_1\)-C\(_{20}\))hydrocarbyl, with the proviso that \( R^1 \) and \( R^2 \) do not include an amino-functional group. The variable \( R^3 \) is at each occurrence independently at least one of straight chain, branched, and cyclic, substituted or unsubstituted (C\(_1\)-C\(_{20}\))hydrocarbylene optionally interrupted by 1-3 oxygen atoms. In addition, DP is about 1 to about 500.

[0004] In various embodiments, the present invention provides a hydroxylamine-terminated polysiloxane having the structure \( E-R^3-\left[\text{SiR}^1\text{R}^2\text{O}\right]_{DP}\text{SiR}^1\text{R}^2-R^3-E \). The variable \( E \) can be a monovalent hydroxylamino functional group -Y-R\(^6\). The variable \( Y \) can be one of:
The variable $R^6$ can be $-NR^7R^8$. Each of $R^7$ and $R^8$ at each occurrence can be independently H or at least one of straight-chain, branched, and cyclic, substituted or unsubstituted $(C_1-C_{30})$ hydrocarbyl optionally substituted with at least one hydroxyl. Each of $R^1$ and $R^2$ can independently be methyl or ethyl. The variable $R^3$ can be $-R^4\cdot A\cdot R^5\cdot$. The variable $A$ can be $-\cdot O\cdot$ or a bond. The variable $R^4$ can be a $(C_2-C_{10})$ hydrocarbylene. The variable $R^5$ can be a bond or a substituted or unsubstituted $(C_1-C_{10})$ hydrocarbylene. In addition, the variable $D$ can be about 1 to about 400.

[0005] In various embodiments, the present invention provides a method of making the hydroxylamine-terminated polysiloxane having the structure $E\cdot R^3\cdot [SiR^1R^2O]_D\cdot SiR^1R^2\cdot R^3\cdot E$. The method includes obtaining or providing an epoxy-terminated diorganopolysiloxane. The method also includes reacting the epoxy-terminated diorganopolysiloxane with a primary or secondary amine, to provide the hydroxylamine-terminated polysiloxane.

[0006] In various embodiments, the present invention provides a method of making the hydroxylamine-terminated polysiloxane having the structure $E\cdot R^3\cdot [SiR^1R^2O]_D\cdot SiR^1R^2\cdot R^3\cdot E$. The method includes obtaining or providing a hydroxylamino-terminated 1,1,3,3-tetraorganodisiloxane. The method also includes reacting the hydroxylamino-terminated tetraorganodisiloxane with a cyclopolydiorganosiloxane, to provide the hydroxylamine-terminated polysiloxane.

[0007] In various embodiments, the present invention provides a hair care composition including the hydroxylamine-terminated polysiloxane, and methods of hair treatment including contacting at least one mammalian hair with the hair care composition. In some embodiments, the present invention provides a textile treatment composition including the hydroxylamine-terminated polysiloxane, and methods of textile treatment including contacting a textile with the textile treatment composition. In some embodiments, the present invention provides an aqueous emulsion comprising the hydroxylamine-terminated
polysiloxane, and methods of treating at least one mammalian hair, textile, or fiber with a composition including the emulsion.

[0008] Various embodiments of the present invention provide advantages over other amine-functional polysiloxanes, at least some of which are unexpected. For example, in various embodiments, the hydroxylamine-terminated polysiloxane lessens disadvantages of using amine groups on polysiloxanes. For example, by only having amine groups located at the termini of the polymer, the hydroxylamine-terminated polysiloxanes of the present invention can have fewer amine-functional groups than polysiloxanes having pendant amine-functional groups, since the number of pendant amine-functional groups on a given polysiloxane can be difficult to control and can often be greater than two. In another example, by having amine-functional groups only located at terminal positions on the polysiloxane, various embodiments of hydroxylamine-terminated polysiloxane of the present invention can use amine-functional groups in a more efficient manner than amine-functional polysiloxanes having amine-functional groups at pendant locations. Pendant locations can be sterically hindered due to the presence of the polysiloxane backbone extending in two directions from the point of substitution, making it more difficult for pendant amine groups to interact with other functional groups such as those found on fibers, textiles, and hair. In contrast, terminal amine-functional groups can be less sterically hindered and can more easily interact with other functional groups. Therefore, for a given total amount of desired interaction between amine-functional groups on a polysiloxane molecule and other functional groups on a substrate, a smaller number of terminal amine-functional groups can produce the desired interaction, as compared to the number of pendant amine-functional groups, or the number of combined pendant and terminal amine-functional groups, that can produce the same amount of interaction.

[0009] In some embodiments, by using hydrosilylation chemistry in conjunction with the amine-epoxide chemistry, substantially all polysiloxane chains have two amine-functional groups; in contrast, in pendant amine-functionalized polysiloxanes made via equilibration methods, frequently some polymer strands have no amine functionalization and thus are wasted and cannot interact effectively with the desired functional groups on a substrate such as hair, textiles, or fiber. In various embodiments, the hydroxylamine-terminated polysiloxane can be more economically produced than hydroxylamine-terminated copolymers having polysiloxane repeating units therein, due to the extra steps required to introduce non-siloxane units into the polymer. In some examples, as compared to polysiloxanes having amine-functionality but no hydroxyl-functionality, the hydroxylamine-functional polysiloxane produces superior results in the treatment of various substrates, such as hair, fiber, or textiles, due to the presence of the terminal-hydroxyl functionality.
For example, the hydroxyl group can produce a greater degree of softness than an amine functionality alone.

DETAILED DESCRIPTION OF THE INVENTION

[0010] Reference will now be made in detail to certain embodiments of the disclosed subject matter. While the disclosed subject matter will be described in conjunction with the enumerated claims, it will be understood that the exemplified subject matter is not intended to limit the claims to the disclosed subject matter.

[0011] Values expressed in a range format should be interpreted in a flexible manner to include not only the numerical values explicitly recited as the limits of the range, but also to include all the individual numerical values or sub-ranges encompassed within that range as if each numerical value and sub-range is explicitly recited. For example, a range of “about 0.1% to about 5%” or “about 0.1% to 5%” should be interpreted to include not just about 0.1% to about 5%, but also the individual values (e.g., 1%, 2%, 3%, and 4%) and the sub-ranges (e.g., 0.1% to 0.5%, 1.1% to 2.2%, 3.3% to 4.4%) within the indicated range. The statement “about X to Y” has the same meaning as “about X to about Y,” unless indicated otherwise. Likewise, the statement “about X, Y, or about Z” has the same meaning as “about X, about Y, or about Z,” unless indicated otherwise.

[0012] In this document, the terms “a,” “an,” or “the” are used to include one or more than one unless the context clearly dictates otherwise. The term “or” is used to refer to a nonexclusive “or” unless otherwise indicated. In addition, it is to be understood that the phraseology or terminology employed herein, and not otherwise defined, is for the purpose of description only and not of limitation. Any use of section headings is intended to aid reading of the document and is not to be interpreted as limiting; information that is relevant to a section heading may occur within or outside of that particular section. Furthermore, all publications, patents, and patent documents referred to in this document are incorporated by reference herein in their entirety, as though individually incorporated by reference. In the event of inconsistent usages between this document and those documents so incorporated by reference, the usage in the incorporated reference should be considered supplementary to that of this document; for irreconcilable inconsistencies, the usage in this document controls.

[0013] In the methods of manufacturing described herein, the steps can be carried out in any order without departing from the principles of the invention, except when a temporal or operational sequence is explicitly recited.

[0014] Furthermore, specified steps can be carried out concurrently unless explicit claim language recites that they be carried out separately. For example, a claimed step of doing X and a claimed step of doing Y can be conducted simultaneously within a single operation, and the resulting process will fall within the literal scope of the claimed process.
[0015] The term “about” as used herein can allow for a degree of variability in a value or range, for example, within 10%, within 5%, or within 1% of a stated value or of a stated limit of a range.

[0016] The term “substantially” as used herein refers to a majority of, or mostly, as in at least about 50%, 60%, 70%, 80%, 90%, 95%, 96%, 97%, 98%, 99%, 99.5%, 99.9%, 99.99%, or at least about 99.999% or more.

[0017] The term “organic group” as used herein refers to but is not limited to any carbon-containing functional group.

[0018] The term “substituted” as used herein refers to an organic group as defined herein or molecule in which one or more hydrogen atoms contained therein are replaced by one or more non-hydrogen atoms. The term “functional group” or “substituent” as used herein refers to a group that can be or is substituted onto a molecule, or onto an organic group. Examples of substituents or functional groups include, but are not limited to, a halogen (e.g., F, Cl, Br, and I); an oxygen atom in groups such as hydroxyl groups, alkoxy groups, aryloxy groups, aralkyloxy groups, oxo(carbonyl) groups, carboxyl groups including carboxylic acids, carboxylates, and carboxylate esters; a sulfur atom in groups such as thiol groups, alkyl and aryl sulfide groups, sulfoxide groups, sulfone groups, sulfonyl groups, and sulfonamide groups; a nitrogen atom in groups such as amines, hydroxylamines, nitriles, nitro groups, N-oxides, hydrazides, azides, and enamines; and other heteroatoms in various other groups. Non-limiting examples of substituents J that can be bonded to a substituted carbon (or other) atom include F, Cl, Br, I, OR', OC(O)N(R')2, CN, NO, NO2, ONO2, azido, CF3, OCF3, R' O (oxo), S (thiono), C(O), S(O), methylenedioxy, ethylenedioxy, N(R')2, SR', SOR', SO2R', SO2N(R')2, SO3R', C(O)R', C(O)C(O)R', C(O)CH2C(O)R', C(S)R', C(O)OR', OC(O)R', C(O)N(R')2, OC(O)N(R')2, C(S)N(R')2, (CH2)0-2N(R')C(O)R', (CH2)0-2N(R')N(R')2, N(R')N(R')C(O)R', N(R')N(R')C(O)OR', N(R')N(R')CON(R')2, N(R')SO2R', N(R')SO2N(R')2, N(R')C(O)OR', N(R')C(O)R', N(R')C(S)R', N(R')C(O)N(R')2, N(R')C(S)N(R')2, N(COR')COR', N(OR')R', C(=NH)N(R')2, C(O)N(OR')R', or C(=NOR')R' wherein R' can be hydrogen or a carbon-based moiety, and wherein the carbon-based moiety can itself be further substituted; for example, wherein R' can be hydrogen, alkyl, acyl, cycloalkyl, aryl, aralkyl, heterocyclyl, heteroaryl, or heteroaryalkyl, wherein any alkyl, acyl, cycloalkyl, aryl, aralkyl, heterocyclyl, heteroaryl, or heteroaryalkyl or R' can be independently mono- or multi-substituted with J; or wherein two R' groups bonded to a nitrogen atom or to adjacent nitrogen atoms can together with the nitrogen atom or atoms form a heterocyclyl, which can be mono- or independently multi-substituted with J.
[0019] The term “alkyl” as used herein refers to straight chain and branched alkyl groups and cycloalkyl groups having from 1 to 40 carbon atoms, 1 to about 20 carbon atoms, 1 to 12 carbons or, in some embodiments, from 1 to 8 carbon atoms. Examples of straight chain alkyl groups include those with from 1 to 8 carbon atoms such as methyl, ethyl, n-propyl, n-butyl, n-pentyl, n-hexyl, n-heptyl, and n-octyl groups. Examples of branched alkyl groups include, but are not limited to, isopropyl, iso-butyl, sec-butyl, t-butyl, neopentyl, isopentyl, and 2,2-dimethylpropyl groups. As used herein, the term “alkyl” encompasses n-alkyl, isoalkyl, and anteisoalkyl groups as well as other branched chain forms of alkyl. Representative substituted alkyl groups can be substituted one or more times with any of the groups listed herein, for example, amino, hydroxy, cyano, carboxy, nitro, thio, alkoxy, and halogen groups.

[0020] The term “alkenyl” as used herein refers to straight and branched chain and cyclic alkyl groups as defined herein, except that at least one double bond exists between two carbon atoms. Thus, alkenyl groups have from 2 to 40 carbon atoms, or 2 to about 20 carbon atoms, or 2 to 12 carbons or, in some embodiments, from 2 to 8 carbon atoms. Examples include, but are not limited to vinyl, -CH=CH(CH3), -CH=C(CH3)2, -C(CH3)=CH2, -C(CH3)=CH(CH3), -C(CH2CH3)=CH2, cyclohexenyl, cyclopentenyl, cyclohexadienyl, butadienyl, pentadienyl, and hexadienyl among others.

[0021] The term “alkynyl” as used herein refers to straight and branched chain alkyl groups, except that at least one triple bond exists between two carbon atoms. Thus, alkenyl groups have from 2 to 40 carbon atoms, 2 to about 20 carbon atoms, or from 2 to 12 carbons or, in some embodiments, from 2 to 8 carbon atoms. Examples include, but are not limited to -C=CH, -C=C(CH3), -C=C(CH2CH3), -CH2C=CH, -CH2C=C(CH3), and -CH2C=C(CH2CH3) among others.

[0022] The term “acyl” as used herein refers to a group containing a carbonyl moiety wherein the group is bonded via the carbonyl carbon atom. The carbonyl carbon atom is also bonded to another carbon atom, which can be part of an alkyl, aryl, aralkyl cycloalkyl, cycloalkylalkyl, heterocyclyl, heterocyclylalkyl, heteroaryl, heteroarylalkyl group or the like. In the special case wherein the carbonyl carbon atom is bonded to a hydrogen, the group is a “formyl” group, an acyl group as the term is defined herein. An acyl group can include 0 to about 12-20 or 12-40 additional carbon atoms bonded to the carbonyl group. An acyl group can include double or triple bonds within the meaning herein. An acryloyl group is an example of an acyl group. An acyl group can also include heteroatoms within the meaning here. A nicotinyl group (pyridyl-3-carbonyl) group is an example of an acyl group within the meaning herein. Other examples include acetyl, benzoyl, phenylacetyl, pyridylacetyl, cinnamoyl, and acryloyl groups and the like. When the group containing the
carbon atom that is bonded to the carbonyl carbon atom contains a halogen, the group is termed a "haloacyl" group. An example is a trifluoroacetyl group.

[0023] The term "cycloalkyl" as used herein refers to cyclic alkyl groups such as, but not limited to, cyclopropyl, cyclobutyl, cyclopentyl, cyclohexyl, cycloheptyl, and cyclooctyl groups. In some embodiments, the cycloalkyl group can have 3 to about 8-12 ring members, whereas in other embodiments the number of ring carbon atoms range from 3 to 4, 5, 6, or 7. Cycloalkyl groups further include polycyclic cycloalkyl groups such as, but not limited to, norbornyl, adamantyl, bornyl, camphenyl, isocamphenyl, and carenyl groups, and fused rings such as, but not limited to, decalinyl, and the like. Cycloalkyl groups also include rings that are substituted with straight or branched chain alkyl groups as defined herein. Representative substituted cycloalkyl groups can be mono-substituted or substituted more than once, such as, but not limited to, 2,2-, 2,3-, 2,4- 2,5- or 2,6-disubstituted cyclohexyl groups or mono-, di- or tri-substituted norbornyl or cycloheptyl groups, which can be substituted with, for example, amino, hydroxy, cyano, carboxy, nitro, thio, alkoxy, and halogen groups. The term "cycloalkenyl" alone or in combination denotes a cyclic alkenyl group.

[0024] The term "aryl" as used herein refers to cyclic aromatic hydrocarbons that do not contain heteroatoms in the ring. Thus aryl groups include, but are not limited to, phenyl, azulenyl, heptalenyl, biphenyl, indacenyl, fluorenyl, phenanthrenyl, triphenylene, pyrenyl, naphthalenyl, chrysene, biphenylene, anthracenyl, and naphthyl groups. In some embodiments, aryl groups contain about 6 to about 14 carbons in the ring portions of the groups. Aryl groups can be un-substituted or substituted, as defined herein. Representative substituted aryl groups can be mono-substituted or substituted more than once, such as, but not limited to, 2-, 3-, 4-, 5-, or 6-substituted phenyl or 2-8 substituted naphthyl groups, which can be substituted with carbon or non-carbon groups such as those listed herein.

[0025] The term "aralkyl" as used herein refers to alkyl groups as defined herein in which a hydrogen or carbon bond of an alkyl group is replaced with a bond to an aryl group as defined herein. Representative aralkyl groups include benzyl and phenylethyl groups and fused (cycloalkylaryl)alkyl groups such as 4-ethyl-indanyl. Aralkenyl group are alkenyl groups as defined herein in which a hydrogen or carbon bond of an alkyl group is replaced with a bond to an aryl group as defined herein.

[0026] The term "heterocyclyl" as used herein refers to aromatic and non-aromatic ring compounds containing 3 or more ring members, of which, one or more is a heteroatom such as, but not limited to, N, O, and S. Thus a heterocyclyl can be a cycloheteroalkyl, or a heteroaryl, or if polycyclic, any combination thereof.
[0027] The term “heteroaryl” as used herein refers to aromatic ring compounds containing 5 or more ring members, of which, one or more is a heteroatom such as, but not limited to, N, O, and S; for instance, heteroaryl rings can have 5 to about 8-12 ring members. A heteroaryl group is a variety of a heterocyclyl group that possesses an aromatic electronic structure.

[0028] The term “heterocyclylalkyl” as used herein refers to alkyl groups as defined herein in which a hydrogen or carbon bond of an alkyl group as defined herein is replaced with a bond to a heterocyclyl group as defined herein. Representative heterocyclyl alkyl groups include, but are not limited to, furan-2-yl methyl, furan-3-yl methyl, pyridine-3-yl methyl, tetrahydrofuran-2-yl ethyl, and indol-2-yl propyl.

[0029] The term “heteroarylalkyl” as used herein refers to alkyl groups as defined herein in which a hydrogen or carbon bond of an alkyl group is replaced with a bond to a heteroaryl group as defined herein.

[0030] The term “alkoxy” as used herein refers to an oxygen atom connected to an alkyl group, including a cycloalkyl group, as are defined herein. Examples of linear alkoxy groups include but are not limited to methoxy, ethoxy, propoxy, butoxy, pentyloxy, hexyloxy, and the like. Examples of branched alkoxy include but are not limited to isopropoxy, sec-butoxy, tert-butoxy, isopentylxoxy, isohexyloxy, and the like. Examples of cyclic alkoxy include but are not limited to cyclopropyloxy, cyclobutyloxy, cyclopentylxoxy, cyclohexyloxy, and the like. An alkoxy group can include one to about 12-20 or about 12-40 carbon atoms bonded to the oxygen atom, and can further include double or triple bonds, and can also include heteroatoms. For example, an allyloxy group is an alkoxy group within the meaning herein. A methoxyethoxy group is also an alkoxy group within the meaning herein, as is a methylenedioxy group in a context where two adjacent atoms of a structures are substituted therewith.

[0031] The term “amine” as used herein refers to primary, secondary, and tertiary amines having, e.g., the formula N(group)_3 wherein each group can independently be H or non-H, such as alkyl, aryl, and the like. Amines include but are not limited to R-NH₂, for example, alkylamines, arylamines, alkylarylamines; R₂NH wherein each R is independently selected, such as dialkylamines, diarylamines, aralkylamines, heterocyclylamines and the like; and R₃N wherein each R is independently selected, such as trialkylamines, dialkylarylamines, alkylidarylamines, triarylamines, and the like. The term ”amine” also includes ammonium ions as used herein.

[0032] The term “amino group” as used herein refers to a substituent of the form -NH₂, -NHR, -NR₂, -NR₃⁺, wherein each R is independently selected, and protonated forms of each, except for -NR₃⁺, which cannot be protonated. Accordingly, any compound
substituted with an amino group can be viewed as an amine. An "amino group" within the meaning herein can be a primary, secondary, tertiary or quaternary amino group. An "alkylamino" group includes a monoalkylamino, dialkylamino, and trialkylamino group.

[0033] The terms "halo" or "halogen" or "halide", as used herein, by themselves or as part of another substituent mean, unless otherwise stated, a fluorine, chlorine, bromine, or iodine atom, preferably, fluorine, chlorine, or bromine.

[0034] The term "haloalkyl" group, as used herein, includes mono-halo alkyl groups, poly-halo alkyl groups wherein all halo atoms can be the same or different, and per-halo alkyl groups, wherein all hydrogen atoms are replaced by halogen atoms, such as fluoro.

Examples of haloalkyl include trifluoromethyl, 1,1-dichloroethyl, 1,2-dichloroethyl, 1,3-dibromo-3,3-difluoropropyl, perfluorobutyl, and the like.

[0035] The term "epoxide-containing" as used herein refers to a functional group in which an oxygen atom, the epoxy substituent, is directly attached to two adjacent carbon atoms of a carbon chain or ring system. Examples of epoxide-containing functional groups include, but are not limited to, 2,3-epoxypropyl, 3,4-epoxybutyl, 4,5-epoxypentyl, 2-glycidoxyethyl, 3-glycidoxypropyl, 4-glycidoxybutyl, 2-(3,4-epoxy-3-methyllohexyl)ethyl, 3-(3,4-epoxy-3-methyllohexyl)propyl, 2-(3,4-epoxy-3-methyllohexyl)-2-methylethyl, 2-(2,3-epoxy-3-methyllohexyl)ethyl, and 3-(2,3-epoxylopetlyl)propyl.

[0036] The term "monovalent" as used herein refers to a substituent connecting via a single bond to a substituted molecule. When a substituent is monovalent, such as, for example, F or Cl, it is bonded to the atom it is substituting by a single bond.

[0037] The term "hydrocarbon" as used herein refers to a functional group or molecule that includes carbon and hydrogen atoms. The term can also refer to a functional group or molecule that normally includes both carbon and hydrogen atoms but wherein all the hydrogen atoms are substituted with other functional groups.

[0038] The term "resin" as used herein refers to polysiloxane material of any viscosity that includes at least one siliocane monomer that is bonded via a Si-O-Si bond to three or four other siloxane monomers. In one example, the polysiloxane material includes T or Q groups, as defined herein.

[0039] The term "number-average molecular weight" as used herein refers to the ordinary arithmetic mean of the molecular weight of individual molecules in a sample. It is defined as the total weight of all molecules in a sample divided by the total number of molecules in the sample. Experimentally, the number average molecular weight (M_n) is determined by analyzing a sample divided into molecular weight fractions of species i having n_i molecules of molecular weight M_i through the formula \( M_n = \sum M_i n_i / \Sigma n_i \). The number average molecular weight can be measured by a variety of well-known methods including gel permeation chromatography, spectroscopic end group analysis and osmometry.
[0040] The term “oligomer” as used herein refers to a molecule having an intermediate relative molecular mass, the structure of which essentially includes a small plurality of units derived, actually or conceptually, from molecules of lower relative molecular mass. A molecule having an intermediate relative mass can be a molecule that has properties that vary with the removal of one or a few of the units. The variation in the properties that results from the removal of the one of more units can be a significant variation.

[0041] The term “solvent” as used herein refers to a liquid that can dissolve a solid, liquid, or gas. Nonlimiting examples of solvents are silicones, organic compounds, water, alcohols, ionic liquids, and supercritical fluids.

[0042] The term “room temperature” as used herein refers to a temperature of about 15 °C to 28 °C.

[0043] The term “thermoplastic polymer” as used herein refers to a polymer that has the property of converting to a fluid (flowable) state when heated and of becoming rigid (nonflowable) when cooled.

[0044] The term “coating” as used herein refers to a continuous or discontinuous layer of material on the coated surface, wherein the layer of material can penetrate the surface and can fill areas such as pores, wherein the layer of material can have any three-dimensional shape, including a flat or curved plane. In one example, a coating can be formed on one or more surfaces, any of which may be porous or nonporous, by immersion in a bath of coating material.

[0045] As used herein, the term “hydrocarbyl” refers to a functional group derived from a straight chain, branched, or cyclic hydrocarbon, such as an alkyl, alkenyl, alkynyl, aryl, cycloalkyl, acyl, or a combination thereof. Hydroxylamine-terminated polysiloxane.

[0046] In various embodiments, the present invention provides a hydroxylamine-terminated polysiloxane having the structure E-R₃-[SiR¹R²O]DPSiR¹R²-R³-E. When DP is greater than 1, the structure E-R₃-[SiR¹R²O]DPSiR¹R²-R³-E represents one or more disiloxane units having substituents R¹ and R² (e.g., SiR¹R²O₂/₃) terminated by monosiloxane units having substituents R¹, R², and –R³-E substituents (e.g., E-R³-SiR¹R²O₁/₂). When DP is 1, the structure E-R₃-[SiR¹R²O]DPSiR¹R²-R³-E represents two monosiloxane units having substituents R¹, R², and –R³-E substituents (e.g., E-R³-SiR¹R²O₁/₂). The variable E is a monovalent hydroxylamino functional group. Each of R¹ and R² is at each occurrence independently at least one of straight chain, branched, and cyclic, substituted or unsubstituted (C₁-C₂₀)hydrocarbyl, with the proviso that R¹ and R² do not include an amino-functional group. In some embodiments, each of R¹ and R² can be substituted or
unsubstituted \((C_1-C_{10})\)alkyl. For example, each of \(R^1\) and \(R^2\) can be methyl, ethyl, or propyl. The variable \(R^3\) is at each occurrence independently at least one of straight chain, branched, and cyclic, substituted or unsubstituted \((C_1-C_{20})\)hydrocarbylene optionally interrupted by 1-3 oxygen atoms. The variable DP can be about 1-500, 1-400, 50-500, 75-250, 1-250, 1-220, 10-250, 20-250, 50-250, or about 100-220. Embodiments of the present invention encompass a plurality of hydroxylamine-terminated polysiloxanes having the structure \(E-R^3-[SiR^1R^2O]_DPSiR^1R^2-R^3-E\), a single polymer having the structure, and any composition including the same.

[0047] In some embodiments, the variable \(R^3\) is \(-R^4-A-R^5-\). The variable \(A\) can be \(-O-\) or a bond. The variable \(R^4\) can be at least one of straight-chain, cyclic, and branched, substituted or unsubstituted \((C_2-C_{10})\)hydrocarbylene. The variable \(R^5\) can be a bond or at least one of straight-chain, cyclic, and branched, substituted or unsubstituted \((C_1-C_{10})\)hydrocarbylene. When adjacent substituents are each a bond, e.g., \(R^5\) and \(A\), the two adjacent substituents together can form a single bond. In some embodiments, each of \(R^4\) and \(R^5\) can be independently \((C_2-C_{10})\)alkylene. In some examples, wherein \(R^4\) can be propylene. In some examples, \(A\) is \(-O-\). In some examples, \(R^5\) is methylene.

[0048] In some examples, in the structure \(E-R^3-[SiR^1R^2O]_DPSiR^1R^2-R^3-E\), \(E\) can be \(-Y-R^6\). The variable \(Y\) can be a divalent ring-opened epoxide. The variable \(R^6\) can be \(-NR^7R^8\). Each of \(R^7\) and \(R^8\) at each occurrence can be independently \(H\) or at least one of branched, straight-chain, and cyclic, substituted or unsubstituted \((C_1-C_{30})\)hydrocarbyl optionally substituted with at least one hydroxyl. At least one, or both, of \(R^7\) and \(R^8\) can be hydroxyl-substituted. In some examples, each of \(R^7\) and \(R^8\) at each occurrence can be at least one of a branched, straight-chain, and cyclic unsubstituted \((C_1-C_{30})\)hydrocarbyl optionally substituted with at least one hydroxyl, provided that at least one of \(R^7\) and \(R^8\) is hydroxyl-substituted; in other embodiments, neither or \(R^7\) nor \(R^8\) are hydroxyl-substituted. The variables \(R^7\) and \(R^8\) can each independently be a branched, cyclic, or straight chain \((C_1-C_{30})\)alkyl group. The variable \(R^7\) can be a branched, cyclic, or straight chain \((C_1-C_{30})\)alkyl group, and the variable \(R^8\) can be a hydroxyl-substituted branched, cyclic, or straight chain \((C_1-C_{30})\)alkyl group. The variables \(R^7\) and \(R^8\) can each independently be hydroxyl-substituted branched, cyclic, or straight chain \((C_1-C_{30})\)alkyl group. In some examples, \(R^7\) can be methyl and \(R^8\) can be hydroxylethyl. In some examples, \(R^7\) can be
methyl and R⁸ can be octadecyl. In some examples, R⁷ and R⁸ can be 2-hydroxypropanol.

[0049] In some embodiments, Y is

5 In some examples, Y is

In some examples, R⁶ is a primary amine, a secondary amine, or a tertiary amine. In some examples, R⁶ can be (CH₃)HN-, (CH₃)₂N-, (CH₃CH₂)HN-, (CH₃CH₂)₂N-, and (HOCH₂CH₂)₂N-. In some examples, R⁶ can include cyclic amines, such as pyrrolidine, piperidine, morpholine, 3-pyrrolidinol, 2,5-dimethylpyrrolidine, 1-methylpiperazine, 4-hydroxypiperidine, N-(2-hydroxyethyl)piperazine, 2,6-dimethylpiperidine, 1-ethylpiperazine, 1-amino-4-methylpiperazine, and isoindoline. The variable R⁶ can include other organic functional groups, such as amino, hydroxyl, ether, amido, and carboxyl groups. The hydroxyl functionality of the hydroxylamine-functional group can be at least in part from the hydroxyl formed by opening the epoxide. Optionally, in addition, the hydroxylamine-functional group can include other hydroxyl functionality. For example, R⁶ can be

20 In some examples, R⁶ is
[0050] In some embodiments, in the structure $E - R^3 - [SiR^1R^2O]_DP SiR^1R^2 - R^3 - E$, $-R^3 - E$ is

The variable $R^6$ can be $-NR^7 R^8$. Each of $R^7$ and $R^8$ at each occurrence can be

independently at least one of straight-chain, branched, and cyclic, substituted or

unsubstituted (C$_1$-C$_{30}$)hydrocarbyl, wherein at least one of $R^7$ and $R^8$ is hydroxyl-

substituted.

[0051] The hydroxylamine-terminated polysiloxane can have any suitable properties,

consistent with the structure as described herein. The hydroxylamine-terminated

polysiloxane can have any suitable viscosity. For example, in some embodiments, the

hydroxylamine-terminated polysiloxane can have a viscosity at room temperature of about

10 cP to 30,000 cP, 30 cP to 20,000 cP, or about 300 cP to 18,000 cP. In some examples,

the clarity of the polymer can be transparent or hazy. In various examples, the color can

be clear to yellow- or orange-tinted.

[0052] In any embodiment described herein, the amine groups can be present in a

quaternary ammonium form, for example as resulting from addition of acid or via the use of

common quaternizing agents such as alkyl halides, benzyl halides, alkyl sulfates, aryl

sulfates, or chloroacetate esters.

Method of making the hydroxylamine-terminated polysiloxane.

[0053] In various embodiments, the present invention provides a method of making the

hydroxylamine-terminated polysiloxane. The method can be any suitable chemical

synthetic method that produces the hydroxylamine-terminated polysiloxane as described

herein.

[0054] For example, a method of making the hydroxylamine-terminated polysiloxane can

include obtaining or providing an epoxy-terminated diorganopolysiloxane. The method can

also include reacting the epoxy-terminated diorganopolysiloxane with a primary or

secondary amine, to provide the hydroxyl-amine terminated polysiloxane. The epoxy-

terminated diorganopolysiloxane can have the structure $R^9 - R^3 - [SiR^1R^2O]_DP SiR^1R^2 - R^3 -

R^9$. The variable $R^9$ can be a monovalent epoxide-containing functional group. The

epoxide-terminated diorganopolysiloxane can be a product of a hydrosilation reaction
between an Si-H-terminated polydiorganosiloxane and an alkenylepoxide or alkynylepoxide.

[0055] In another embodiment, the method of making the hydroxylamine-terminated polysiloxane includes obtaining or providing a hydroxylamino-terminated 1,1,3,3-tetraorganodisiloxane. The method also can include reacting the hydroxylamino-terminated 1,1,3,3-tetraorganodisiloxane with a cyclopolysilorganosiloxane, to provide the hydroxylamine-terminated polysiloxane. The hydroxylamino-terminated 1,1,3,3-tetraorganodisiloxane can have the following structure:

\[ E \cdot R^3 \cdot SiR^1 R^2 OSiR^1 R^2 \cdot R^3 \cdot E. \]

The hydroxylamino-terminated 1,1,3,3-tetraorganodisiloxane can be the product of a reaction between a primary or secondary amine and an epoxide-terminated 1,1,3,3-tetraorganodisiloxane. In some examples, the epoxide-terminated 1,1,3,3-tetraorganodisiloxane can have the structure \( R^9 \cdot R^3 \cdot SiR^1 R^2 OSiR^1 R^2 \cdot R^3 \cdot R^9 \). The variable \( R^9 \) can be a monovalent epoxide-containing functional group. The epoxide-terminated 1,1,3,3-tetraorganodisiloxane can be the product of an Si-H-terminated 1,1,3,3-tetraorganodisiloxane and an alkenylepoxide or alkynylepoxide.

[0056] The alkenylepoxide or alkynylepoxide described herein can be any suitable alkenylepoxide or alkynylepoxide. For example, the alkenylepoxide or alkynylepoxide can have the structure \( R^9 \cdot R^3 \)’, wherein \( R^3 \)’ is \(-R^5 \cdot A \cdot R^4 \)’. The variable \( R^4 \)’ can be a \( C_2\)-\( C_{10} \)hydrocarbyl corresponding to \( R^4 \) and having at least one aliphatic unsaturated carbon-carbon bond, for example \( H_2C=CH_{-} \), \( H_2C=CHCH_{2_{-}} \), \( H_2C=C(\text{CH}_3)\text{CH}_2_{-} \), \( H_2C=\text{CHCH}_2\text{CH}_2_{-} \), \( H_2C=\text{CHCH}_2\text{CH}_2\text{CH}_2_{-} \), \( H_2C=\text{CHCH}_2\text{CH}_2\text{CH}_2\text{CH}_2_{-} \), \( \text{HC} = \text{CC} \cdot \text{CH}_2_{-} \), \( \text{HC} = \text{CC}(\text{CH}_3)_{-} \), \( \text{HC} = \text{CC}(\text{CH}_3)_{2_{-}} \), and \( \text{HC} = \text{CC}(\text{CH}_3)_{2\cdot} \text{CH}_2_{-} \). The variable \( R^9 \) can be an epoxide-containing group. In some embodiments, \( R^9 \) can be one of

\[
\begin{align*}
\text{and} \\
\text{and}
\end{align*}
\]

In various examples, the alkenylepoxide or alkynylepoxide can be allyl glycidyl ether (CAS 106-92-3), vinylcyclohexene oxide (CAS 106-86-5), 5,6-epoxy-1-hexene (CAS 10353-53-4), 9,10-epoxy-1 decene (CAS 85721-25-1), 7,8-epoxy-1-octene (CAS: 19600-63-6), 3,4-
epoxy-1-butene (CAS 930-22-3), 2-methyl-2-vinylxirane (CAS 1838-94-4), glycidyl acrylate, glycidyl methacrylate (CAS 106-91-2), limonene oxide (CAS 1195-92-2), allyloxy-3,4-epoxytricyclo(5.2.1.0 2,6)decane (CAS: 2279-19-8).

[0057] The primary or secondary amine described herein can be any suitable primary or secondary amine. In various examples, the primary or secondary amine can be HNR\textsuperscript{7}R\textsuperscript{8}. Each of R\textsuperscript{7} and R\textsuperscript{8} at each occurrence independently can be H or at least one of straight-chain, branched, and cyclic, substituted or unsubstituted (C\textsubscript{1}-C\textsubscript{30})hydrocarbyl optionally substituted with at least one hydroxyl. For example, the amine can be dimethyamine, dihydroxymethylamine, methylhydroxymethylamine, methyllethylamine,

hydroxymethylhydroxyethylamine, methylhydroxyethylamine (e.g., methylethanolamine), hydroxymethylethylamine, diethylamine, di(hydroxyethyl)amine, hydroxyethyllethylamine, ethylproplamine, hydroxyethylhydroxypropylamine, ethylhydroxypropylamine, hydroxyethylpropylamine, dipropylamine, diisopropylamine, dihydroxyisopropylamine (e.g., diisopropanolamine), hydroxypropylpropylamine, hydroxyisopropylisopropylamine, or di(C\textsubscript{1}-C\textsubscript{30})akyl substituted amines like n-methyloctadecylamine.

Composition including the hydroxylamine-terminated polysiloxane.

[0058] Various embodiments of the present invention provide compositions that include one or more of the hydroxylamine-terminated polysiloxanes described herein. The composition that includes the polysiloxane can be any suitable composition that is used for any suitable purpose consistent with the properties of the polysiloxane or composition including the same. In some embodiments, the present invention provides a personal care composition including the hydroxylamine-terminated polysiloxane described herein. The personal care composition can be a hair care composition.

[0059] The composition can include any suitable amount of the hydroxylamine-terminated polysiloxane. For example, the composition can include about 0.001 wt%, 0.01, 0.1, 1, 2, 3, 4, 5, 10, 15, 20, 30, 40, 50, 60, 70, 80, 85, 90, 95, 96, 97, 98, 99, 99.9, 99.99, or about 99.999 or more wt% of the hydroxylamine-terminated polysiloxane.

[0060] The composition including the hydroxylamine-terminated polysiloxane can be a textile treatment composition. Various embodiments can give a softer feel, a greater smoothness, or other effects, to textiles after contacting under suitable conditions. In some examples, hydroxyl or amino functionalities can aid in anchoring the polymer to a substrate through, for example, hydrogen bonding and van der Waals interactions with the substrate. Hydroxyl and amine functionalities can work together synergistically to improve this interaction.

[0061] The composition including the hydroxylamine-terminated polysiloxane can be an emulsion, such as an aqueous emulsion. In some examples, the hydroxyl-terminated polysiloxane in an emulsion can include at least some amine-groups that are quaternary; in
other examples, the hydroxyl-terminated polysiloxane in an emulsion includes no quaternary amines. The emulsion can have an average particle size of about 0.01 microns to about 1000 microns. As used herein, “emulsion” refers to water continuous emulsions (for example an oil in water type emulsion, or a silicone in water emulsion), oil or silicone continuous emulsions (water in oil emulsions or water in silicone emulsions), or multiple emulsions (water/oil/water, oil/water/oil types, water/silicone/water, or silicone/water/silicone). The hydroxylamine-terminated polysiloxanes of the present disclosure can be emulsified by common mixing techniques. Examples of mixing techniques can include simple stirring, homogenizing, sonicotating, and other mixing techniques known in the art to effect the formation of emulsions. The mixing can be conducted in a batch, semi-continuous, or continuous process.

[0062] The emulsions can be w/o, w/s, or multiple phase emulsions, e.g., using silicone emulsifiers. A water-in-silicone emulsifier can be non-ionic and can be selected from polyoxyalkylene-substituted silicones, silicone alkanolamides, silicone esters and silicone glycosides. Silicone-based surfactants may be used to form such emulsions and are well known in the art, are described for example in US 4,122,029 (Gee et al.), US 5,387,417 (Rentsch), and US 5,811,487 (Schulz et al).

[0063] When the emulsion is an oil-in-water emulsion, it may include common ingredients generally used for preparing emulsions such as but not limited to non-ionic surfactants well known in the art to prepare o/w emulsions. Examples of nonionic surfactants can include polyoxyethylene alkyl ethers, polyoxyethylene alkylphenol ethers, polyoxyethylene lauryl ethers, polyoxyethylene sorbitan monoleates, polyoxyethylene alkyl esters, polyoxyethylene sorbitan alkyl esters, polyethylene glycol, polypropylene glycol, diethylene glycol, ethoxylated trimethylnonanols, and polyoxyalkylene glycol modified polysiloxane surfactants.

Method of using the hydroxylamine-terminated polysiloxane.

[0064] In various embodiments, the present invention provides a method of using the hydroxylamine-terminated polysiloxane. The method can be any suitable method of using the polysiloxane consistent with the structure and properties of the polysiloxane and compositions including the same described herein.

[0065] For example, some embodiments provide a method of treating at least one mammalian hair, textile, or fiber with a composition comprising the hydroxylamine-terminated polysiloxane. In some examples, the composition is an emulsion comprising the hydroxylamine-terminated polysiloxane.

[0066] In some embodiments, the present invention provides a method of fiber or textile treatment. The method can include contacting a textile or fiber with a composition that includes the hydroxylamine-terminated polysiloxane. The amount applied can be a “hand
improving" effective amount of the treatment composition and is applied to the fiber and/or textile by any convenient method. As used herein, "hand" can refer to the softness and smoothness of the fabric. In some examples, the treatment composition can be applied by padding, dipping, spraying or exhausting. When the treatment composition comprises more than one solution, dispersion, or emulsion; the solutions, dispersions, and emulsions can be applied simultaneously or sequentially to the textiles. After the treatment composition is applied to the fiber and/or fabric, it can be dried, for example, by heating. **[0067]** The fiber/textile treatment composition can be applied to the fiber and/or textile at any suitable time, for example during making the fibers or textiles, or later such as during laundering textiles. After application, carriers (if any) can be removed from the treatment composition for example by drying the composition at ambient or elevated temperature. The amount of treatment composition applied to the fibers and textiles is typically sufficient to provide about 0.1 to 15 weight percent of the composition on the fibers and textiles, based on their dry weight, or in an amount of about 0.2 to 5 weight percent based on the dry weight of the fiber or textile. **[0068]** Fibers and textiles that can be treated with the treatment composition are not limited. Examples can include natural fibers such as cotton, silk, linen, and wool; regenerated fibers such as rayon and acetate; synthetic fibers such as polyesters, polyamides, polyacrylonitriles, polyethylenes, and polypropylenes; combinations, and blends thereof. The form of the fibers can include threads, filaments, tows, yarns, woven fabrics, knitted materials, non-woven materials, paper, carpet, and leather.

**Examples**

**[0069]** The present invention can be better understood by reference to the following examples which are offered by way of illustration. The present invention is not limited to the examples given herein. In the Examples, "DP" refers to degree of polymerization. **[0070]** General. The Genapol UD 110 was a polyethylene glycol monoalkyl ether, a C11 oxo alcohol ethoxylate having 11 ethylene oxide units. **Example 1. Direct Route: DIPA Terminated 200 DP Aminosiloxane.**

**Example 1a. Hydrosilylation.**

**[0071]** A three-neck round bottom flask was charged with 22.24 g of reagent grade isopropanol and 195.13 g of SiH terminated siloxane, with a SiH concentration of 134 ppm (201.3 DP) determined via FT-IR. The reaction setup was equipped with a water-cooled condenser, nitrogen sweep, and motorized stirring mechanism for proper agitation, heating mantle, and temperature controller with thermocouple. The flask was purged with nitrogen for five minutes, and then the contents were heated to 80°C. Once the temperature stabilized at 80°C, 3.57 g of allyl glycidyl ether (AGE) was added, which accounts for a 20% molar excess. A 1% solution of Karstedt's catalyst in IPA was added (0.10 g, 5.0 ppm
Pt) as catalyst. An exotherm was observed (+6.1°C), and the reaction temperature was held at 85°C for one hour. The reaction progress was monitored using FT-IR analysis to determine the remaining SiH concentration. Reaction completion ([SiH] < 10 ppm) was verified after two hours, when the SiH concentration was determined to be 7.2 ppm.

Example 1b. Amination.

[0072] The epoxy terminated siloxane intermediate in 10 wt% isopropanol was allowed to cool down to 70°C and 6.15 g (50% molar excess) of an 85% solution of diisopropanolamine (DIPA) in water was added. The amination reaction was allowed to proceed overnight (about 15 hours) at 70°C to ensure complete consumption of the epoxy sites. After holding at temperature, the reaction setup was modified to a simple distillation apparatus. The isopropanol and water was removed from the product by vacuum stripping at 100°C and 20 mmHg for 1 hour. The sample was allowed to cool to less than 60°C prior to breaking the vacuum with nitrogen. Then, the product was decanted into a glass jar. The viscosity of the final product was determined to be 1866 cP at room temperature using a Brookfield DV-II+ rotational viscometer, and the complete consumption of the epoxy groups was verified using 13C NMR analysis by verifying the absence of peaks at chemical shifts of 44 ppm and 51 ppm. The DP of the product was 201.3.

Example 2. Equilibration Route: DIPA 2 DP End-Blocker Synthesis and Equilibration to 200 DP.

Example 2a. Hydrosilylation.

[0073] A three-neck round bottom flask was loaded with 67.52 g of allyl glycidyl ether (AGE), which accounts for a 20% molar excess. An addition funnel was charged with 37.63 g of 1,1,3,3-tetramethyl disiloxane which had an approximate SiH concentration of 13,000 ppm (2.1 DP) determined via FT-IR. The reaction setup was equipped with a water-cooled condenser, nitrogen sweep, and motorized stirring mechanism for proper agitation, heating mantle, and temperature controller with thermocouple. The flask was purged with nitrogen for five minutes, and then the contents were heated to 60°C. Once the temperature has stabilized at 60°C, the siloxane was slowly metered-in drop wise. Once approximately 5-10 wt% of the siloxane was added, a 1% solution of Karstedt's catalyst in IPA was added as necessary to maintain a steady exotherm. Overall, 0.48 g (45 ppm Pt) of catalyst was added to promote the reaction. The temperature was not allowed to increase to greater than 90°C due to the exotherm. Once the addition was complete, the reaction was held at 80°C for one hour. The reaction progress was monitored using FT-IR analysis to determine the remaining SiH concentration. Reaction completion ([SiH] < 10 ppm) was verified two hours after the initial addition, when the SiH concentration was determined to be 5.9 ppm.

Example 2b. Amination with MEA.
A portion (48.59 g) of the epoxy terminated siloxane intermediate (from Example 2a) and 8.60 g of reagent grade isopropanol were loaded into a three-neck round bottom flask equipped with a water-cooled condenser, nitrogen sweep, and motorized stirring mechanism for proper agitation, heating mantle, and temperature controller with thermocouple. The flask was purged with nitrogen for five minutes, and then heated to 70°C. Then, 56.32 g of methylethanolamine (MEA) was added, which accounts for a 100% molar excess relative to the theoretical epoxy sites. The amination reaction was allowed to proceed overnight (about 15 hours) at 70°C to ensure complete consumption of the epoxy sites. After holding at temperature, the reaction setup was modified to a simple distillation apparatus. The isopropanol and excess amine was removed from the product by vacuum stripping at 100°C and 20 mmHg for 1 hour. The sample was allowed to cool to less than 60°C prior to breaking the vacuum with nitrogen and then decanting the product into a glass jar. The viscosity of the final product was determined to be 1,056 cP at room temperature using a Brookfield DV-II+ rotational viscometer, and the complete consumption of the epoxy groups was verified using $^{13}$C NMR analysis by verifying the absence of peaks at chemical shifts of 44 ppm and 51 ppm.

**Example 2c. Amination with DIPA.**

A portion (45.78 g) of the epoxy terminated siloxane intermediate (from Example 2a) and 8.56 g of reagent grade isopropanol was loaded into a three-neck round bottom flask equipped with a water-cooled condenser, nitrogen sweep, and motorized stirring mechanism for proper agitation, heating mantle, and temperature controller with thermocouple. The flask was purged with nitrogen for five minutes, and then heated to 70°C. Then, 56.32 g (50% molar excess) of an 85% solution of di-isopropanolamine (DIPA) in water was added to the flask in a streamwise manner using an addition funnel. Once the addition was complete, the amination reaction was allowed to proceed overnight (about 15 hours) at 70°C to ensure complete consumption of the epoxy sites. After holding at temperature, the reaction setup was modified to a simple distillation apparatus. The isopropanol and water was removed from the product by vacuum stripping at 100°C and 20 mmHg for 1 hour. The sample was allowed to cool to less than 60°C prior to breaking the vacuum with nitrogen and then the product was decanted into a glass jar. The viscosity of the final product was determined to be 13,136 cP at room temperature using a Brookfield DV-II+ rotational viscometer, and the complete consumption of the epoxy groups was verified using $^{13}$C NMR analysis by verifying the absence of peaks at chemical shifts of 44 ppm and 51 ppm.

**Example 2d. Equilibration to 200 DP DIPA Terminated.**

A three-neck round bottom flask was charged with 17.62 g of the 2 DP terminal hydroxylaminosiloxane intermediate (23230-69B). To the flash were added 287.4 g of
decamethylcyclopentasiloxane (245 fluid) and 0.30 g of pelletized KOH. The reaction setup was equipped with a water-cooled condenser, nitrogen sweep, a motorized stirring mechanism for proper agitation, heating mantle, and temperature controller with thermocouple. The flask was purged with nitrogen for five minutes, and then the contents were heated to 150°C. The temperature was held at 150°C for six hours and cooling was initiated. Once the temperature had cooled down to below 85°C, the contents were neutralized with 0.40 g of glacial acetic acid.

[0077] Once the material had reached room temperature, a pressure canister was used to filter the material with 10 μm filter paper and Celite filtration aid. The filtered terminal hydroxylaminosiloxane was loaded into a three-neck round bottom flask equipped with a distillation apparatus setup. The excess cyclics were removed by vacuum stripping at 150°C and application of a vacuum of less than 20 mmHg for 1 hour. The sample was allowed to cool to less than 60°C prior to breaking the vacuum with nitrogen and then decanting the product into a glass jar. The viscosity of the final product was determined to be 3755 cP at room temperature using a Brookfield DV-II+ rotational viscometer. The actual DP was determined to be 149.4 DP using the Varian Inova 400 MHz NMR observing the 29Si nucleus and comparing the ratio of the D-units (chemical shift of δ=-22 ppm) to terminal amino modified M-units (chemical shift of δ=7.5 ppm).


Example 3a. Hydrosilylation.

[0078] A three-neck round bottom flask was charged with 98.92 g of reagent grade isopropanol and 940.7 g of SiH terminated siloxane, with a SiH concentration of 220 ppm (122.6 DP) determined via FT-IR. The reaction setup was equipped with a water-cooled condenser, nitrogen sweep, and motorized stirring mechanism for proper agitation, heating mantle, and temperature controller with thermocouple. The flask was purged with nitrogen for five minutes, and then the contents were heated to 80°C. Once temperature had stabilized at 80°C, 25.95 g of allyl glycidyl ether (AGE) were added, which accounts for a 10% molar excess. A 1% solution of Karstedt’s catalyst in IPA was added (0.39 g, 3.9 ppm Pt) to catalyze the reaction. An exotherm was observed (+7.8°C) and the reaction temperature was held at 85°C for three hours. The reaction progress was monitored using FT-IR analysis to determine the remaining SiH concentration. Reaction completion ([SiH] < 10 ppm) was verified after three hours, when the SiH concentration was determined to be 7.2 ppm. The reaction setup was modified to a simple distillation apparatus. The isopropanol and excess AGE were removed from the product by vacuum stripping at 120°C and 20 mmHg for 2 hours. The sample was allowed to cool to less than 60°C prior to breaking the vacuum with nitrogen and decanting into a glass jar.

Example 3b. Amination with n-methyloctadecylamine
To a three-neck round bottom flask equipped with a water-cooled condenser, nitrogen sweep, and motorized stirring mechanism for proper agitation, heating mantle, and temperature controller with thermocouple was loaded 94.00 g of the epoxy terminated siloxane intermediate (from Example 3a) and 11.11 g of reagent grade isopropanol. To the flask was added 6.00 g of n-methyloctadecylamine (n-MODA), which accounts for a 5% molar excess relative to the theoretical epoxy sites. The flask was purged with nitrogen for five minutes, and then heated to 70°C. The amination reaction was allowed to proceed overnight (about 15 hours) at 70°C to ensure complete consumption of the epoxy sites. After holding at temperature, the reaction setup was modified to a simple distillation apparatus. The isopropanol was removed from the product by vacuum stripping at 100°C and 20 mmHg for 1 hour. The sample was allowed to cool to less than 60°C prior to breaking the vacuum with nitrogen and then decanting the product into a glass jar. The viscosity of the final product was determined to be 435 cP at room temperature using a Brookfield DV-II+ rotational viscometer, and the complete consumption of the epoxy groups was verified using $^{13}$C NMR analysis by verifying the absence of peaks at chemical shifts of 44 ppm and 51 ppm.

Example 3c. Amination with di-isopropanolamine.

Into a three-neck round bottom flask equipped with a water-cooled condenser, nitrogen sweep, and motorized stirring mechanism for proper agitation, heating mantle, and temperature controller with thermocouple was loaded 193.90 g of the epoxy terminated siloxane intermediate (from Example 3a) and 22.94 g of reagent grade isopropanol. Then, 7.17 g of an 85% solution of di-isopropanolamine (DIPA) in water was added to the flask, which accounts for a 10% molar excess relative to the theoretical epoxy sites. The flask was purged with nitrogen for five minutes, and then heated to 70°C. The amination reaction was allowed to proceed overnight (about 15 hours) at 70°C to ensure complete consumption of the epoxy sites. After holding at temperature, the reaction setup was modified to a simple distillation apparatus. The isopropanol was removed from the product by vacuum stripping at 100°C and 20 mmHg for 1 hour. The sample was allowed to cool to less than 60°C prior to breaking the vacuum with nitrogen and then decanting the product into a glass jar. The viscosity of the final product was determined to be 1110 cP at room temperature using a Brookfield DV-II+ rotational viscometer, and the complete consumption of the epoxy groups was verified using $^{13}$C NMR analysis by verifying the absence of peaks at chemical shifts of 44 ppm and 51 ppm.

Example 4. Terminal Amino Siloxane Polymer Emulsion.

Into a 100 Max cup was weighed 57.5 g of terminal amino siloxane polymer (from Example 2d) followed by 3.45 g of Genapol UD 110 and 12 g of DI H2O. The cup was spun for two 30 second cycle at maximum speed using a SpeedMixer DAC 150. Acetic
acid (0.24g) was weighed into the cup. The cup was spun for 30 seconds at maximum speed after which it was diluted with incremental additions of dilution water. The increments began at 10 g and two more additions of dilution water was added, with the total amount of water in the emulsion being 38.81g. The cup was spun in-between each addition of water. The resulting emulsion was an aqueous emulsion of terminal amino polymer siloxane having 57.5% active level. The particle size of the emulsion was 1.33 micron at median diameter measured in volume mode.

[0082] The terms and expressions which have been employed are used as terms of description and not of limitation, and there is no intention that in the use of such terms and expressions of excluding any equivalents of the features shown and described or portions thereof, but it is recognized that various modifications are possible within the scope of the invention claimed. Thus, it should be understood that although the present invention has been specifically disclosed by preferred embodiments and optional features, modification and variation of the concepts herein disclosed may be resorted to by those of ordinary skill in the art, and that such modifications and variations are considered to be within the scope of this invention as defined by the appended claims.

Additional Embodiments.

[0083] The present invention provides for the following exemplary embodiments, the numbering of which is not to be construed as designating levels of importance:

[0084] Embodiment 1 provides a hydroxylamine-terminated polysiloxane having the following structure: E-R³-[SiR¹R²O]DPSiR¹R²-R³-E, wherein E is a monovalent hydroxylamino functional group, each of R¹ and R² is at each occurrence independently at least one of straight chain, branched, and cyclic, substituted or unsubstituted (C₁-C₂₀)hydrocarbyl, with the proviso that R¹ and R₂ do not include an amino-functional group, R³ is at each occurrence independently at least one of straight chain, branched, and cyclic, substituted or unsubstituted (C₁-C₂₀)hydrocarbylene optionally interrupted by 1-3 oxygen atoms, and DP is about 1 to about 500.

[0085] Embodiment 2 provides the polysiloxane of Embodiment 1, wherein each of R¹ and R² are substituted or unsubstituted (C₁-C₁₀)alkyl.

[0086] Embodiment 3 provides the polysiloxane of any one of Embodiments 1-2, wherein each of R¹ and R² are independently methyl or ethyl.

[0087] Embodiment 4 provides the polysiloxane of any one of Embodiments 1-3, wherein R³ is -R⁴-A-R⁵-, wherein A is -O- or a bond, R⁴ is at least one of straight-chain, cyclic, and branched, substituted or unsubstituted (C₂-C₁₀)hydrocarbylene, and R⁵ is a bond or at
least one of straight-chain, cyclic, and branched, substituted or unsubstituted \((C_1-C_{10})\) hydrocarbylene.

[0088] Embodiment 5 provides the polysiloxane of Embodiment 4, wherein each of \(R^4\) and \(R^5\) is independently \((C_2-C_{10})\) alkylene.

[0089] Embodiment 6 provides the polysiloxane of any one of Embodiments 4-5, wherein \(R^4\) is propylene.

[0090] Embodiment 7 provides the polysiloxane of any one of Embodiments 4-6, wherein \(A\) is -O-.

[0091] Embodiment 8 provides the polysiloxane of any one of Embodiments 4-7, wherein \(R^5\) is methylene.

[0092] Embodiment 9 provides the polysiloxane of any one of Embodiments 1-8, wherein \(E\) is -Y-R^6, wherein \(Y\) is a ring-opened epoxide, \(R^6\) is \(-NR^7R^8\), each of \(R^7\) and \(R^8\) at each occurrence is independently \(H\) or at least one of branched, straight-chain, and cyclic, substituted or unsubstituted \((C_1-C_{30})\) hydrocarbyl optionally substituted with at least one hydroxyl.

[0093] Embodiment 10 provides the polysiloxane of Embodiment 9, wherein at least one of \(R^7\) and \(R^8\) is hydroxyl-substituted.

[0094] Embodiment 11 provides the polysiloxane of any one of Embodiments 9-10, wherein each of \(R^7\) and \(R^8\) at each occurrence is at least one of a branched, straight-chain, and cyclic unsubstituted \((C_1-C_{30})\) hydrocarbyl optionally substituted with at least one hydroxyl, provided that at least one of \(R^7\) and \(R^8\) is hydroxyl substituted.

[0095] Embodiment 12 provides the polysiloxane of any one of Embodiments 9-11, wherein \(R^7\) and \(R^8\) are each independently a branched, cyclic, or straight chain \((C_1-C_{30})\) alkyl group.

[0096] Embodiment 13 provides the polysiloxane of any one of Embodiments 9-12, wherein \(R^7\) is a branched, cyclic, or straight chain \((C_1-C_{30})\) alkyl group, and \(R^8\) is a hydroxyl-substituted branched, cyclic, or straight chain \((C_1-C_{30})\) alkyl group.

[0097] Embodiment 14 provides the polysiloxane of any one of Embodiments 9-13, wherein \(R^7\) and \(R^8\) are each independently a hydroxyl-substituted branched, cyclic, or straight chain \((C_1-C_{30})\) alkyl group.

[0098] Embodiment 15 provides the polysiloxane of any one of Embodiments 9-14, wherein \(R^7\) is methyl and \(R^8\) is hydroxylethyl.

[0099] Embodiment 16 provides the polysiloxane of any one of Embodiments 9-15, wherein \(R^7\) is methyl and \(R^8\) is octadecyl.
Embodiment 17 provides the polysiloxane of any one of Embodiments 9-16, wherein R⁷ and R⁸ are 2-hydroxypropanyl.

Embodiment 18 provides the polysiloxane of any one of Embodiments 9-17, wherein Y is

Embodiment 19 provides the polysiloxane of any one of Embodiments 9-18, wherein Y is

Embodiment 20 provides the polysiloxane of any one of Embodiments 9-19, wherein R⁶ is

Embodiment 21 provides the polysiloxane of any one of Embodiments 9-20, wherein R⁶ is

Embodiment 22 provides the polysiloxane of any one of Embodiments 1-21, wherein DP is about 1 to about 400.

Embodiment 23 provides the polysiloxane of any one of Embodiments 1-22, wherein the viscosity at room temperature is about 10 cP to about 30,000 cP.
[00107] Embodiment 24 provides the polysiloxane of any one of Embodiments 1-23, wherein the viscosity at room temperature is about 30 cP to about 20,000 cP.

[00108] Embodiment 25 provides the polysiloxane of any one of Embodiments 1-24, wherein -R^3-E is

\[
\begin{align*}
\text{OH} & \quad \text{O} \quad \text{OH} \\
R^6 & \quad R^8
\end{align*}
\]

wherein R^6 is -NR^7R^8, each of R^7 and R^8 at each occurrence is independently at least one of straight-chain, branched, and cyclic, substituted or unsubstituted (C_1-C_30)hydrocarbyl, wherein at least one of R^7 and R^8 is hydroxyl-substituted.

[00109] Embodiment 26 provides the polysiloxane of any one of Embodiments 1-25, wherein the polysiloxane has the following structure: E-R^3-[SiR^1R^2O]_DpSiR^1R^2-R^3-E,

wherein E is monovalent hydroxylamino functional group -Y-R^6, wherein Y is one of:

\[
\begin{align*}
\text{OH} & \quad \text{OH} & \quad \text{OH} \\
\text{HO} & \quad \text{HO} & \quad \text{HO} \\
\text{HO} & \quad \text{HO} & \quad \text{HO}
\end{align*}
\]

R^6 is -NR^7R^8, and each of R^7 and R^8 at each occurrence is independently H or at least one of straight-chain, branched, and cyclic, substituted or unsubstituted (C_1-C_30)hydrocarbyl.
C$_{30}$)hydrocarbyl optionally substituted with at least one hydroxyl, each of R$^1$ and R$^2$ is methyl, R$^3$ is -R$^4$-A-R$^5$-, wherein A is -O- or a bond, R$^4$ is a (C$_2$-C$_{10}$)hydrocarbylene, and R$^5$ is a bond or a substituted or unsubstituted (C$_1$-C$_{10}$)hydrocarbylene, and DP is about 1 to about 400.

**[00110]** Embodiment 27 provides a method of making the polysiloxane of any one of any one of Embodiments 1-26, the method comprising: obtaining or providing an epoxy-terminated diorganopolysiloxane; reacting the epoxy-terminated diorganopolysiloxane with a primary or secondary amine, to provide the polysiloxane of any one of Embodiments 1-26.

**[00111]** Embodiment 28 provides the method of Embodiment 27, the epoxy-terminated diorganopolysiloxane has the following structure: R$_9$-R$_3$-[SiR$_1$R$_2$O]$_{DP}$SiR$_1$R$_2$-R$_3$-R$_9$ wherein R$_9$ is a monovalent epoxide-containing functional group.

**[00112]** Embodiment 29 provides the method of any one of Embodiments 27-28, wherein the epoxy-terminated diorganopolysiloxane is a product of a hydrosilylation reaction between an Si-H-terminated polydiorganosiloxane and an alkenylepoxide or alkynylepoxide.

**[00113]** Embodiment 30 provides the method of Embodiment 29, wherein the alkenylepoxide or alkynylepoxide has the following structure: R$_9$-R$_3'$, wherein R$_3'$ is -R$_5$-A-R$_4'$, R$_4'$ is an (C$_2$-C$_{10}$)hydrocarbyl corresponding to R$_4$ and having at least one aliphatic unsaturated carbon-carbon bond, and R$_9$ is an epoxide-containing group.

**[00114]** Embodiment 31 provides the method of Embodiment 30, wherein R$_9$ is one of

![Diagram](image)

and

**[00115]** Embodiment 32 provides the method of any one of Embodiments 27-31, wherein the primary or secondary amine is HNR$_7$R$_8$, wherein each of R$_7$ and R$_8$ at each occurrence independently H or at least one of straight-chain, branched, and cyclic, substituted or unsubstituted (C$_1$-C$_{30}$)hydrocarbyl optionally substituted with at least one hydroxyl.
[00116] Embodiment 33 provides a method of making the hydroxylamine-terminated polysiloxane of any one of Embodiments 1-26, the method comprising: obtaining or providing a hydroxylamino-terminated 1,1,3,3-tetraorganodisiloxane; reacting the hydroxylamino-terminated 1,1,3,3-tetraorganodisiloxane with a cyclopolydiorganosiloxane, to provide the polysiloxane of any one of Embodiments 1-26.

[00117] Embodiment 34 provides the method of Embodiment 33, wherein the hydroxylamino-terminated 1,1,3,3-tetraorganodisiloxane has the following structure: E-R^3SIR^1R^2OSIR^1R^2-R^3-E.

[00118] Embodiment 35 provides the method of Embodiment 34, wherein the hydroxylamino-terminated 1,1,3,3-tetraorganodisiloxane is the product of a reaction between a primary or secondary amine and an epoxide-terminated 1,1,3,3-tetraorganodisiloxane.

[00119] Embodiment 36 provides the method of Embodiment 35, wherein the primary or secondary amine is HNR^7R^8, wherein each of R^7 and R^8 at each occurrence independently H or substituted or unsubstituted (C_1-C_10)hydrocarbyl optionally substituted with at least one hydroxyl.

[00120] Embodiment 37 provides the method of any one of Embodiments 35-36, wherein the epoxide-terminated 1,1,3,3-tetraorganodisiloxane has the following structure: R^9-R^3SIR^1R^2OSIR^1R^2-R^3-R^9, wherein R^9 is a monovalent epoxide-containing functional group.

[00121] Embodiment 38 provides the method of any one of Embodiments 35-37, wherein the epoxide-terminated 1,1,3,3-tetraorganodisiloxane is the product of an Si-H-terminated 1,1,3,3-tetraorganodisiloxane and an alkenylepoxide or alkynylepoxide.

[00122] Embodiment 39 provides the method of 38, wherein the alkenylepoxide or alkynylepoxide has the following structure: R^9-R^3', wherein R^3' is -R^5-A-R^4', and R^4' is a (C_2-C_10)hydrocarbyl corresponding to R^4 and having at least one aliphatic unsaturated carbon-carbon bond.

[00123] Embodiment 40 provides the method of any one of Embodiments 37-39, wherein R^9 is one of

\[
\text{Diagram:}
\]
[00124] Embodiment 41 provides a hair care composition comprising the hydroxylamine-termined polysiloxane of any one of Embodiments 1-26.

[00125] Embodiment 42 provides a textile treatment composition comprising the hydroxylamine-termined polysiloxane of any one of Embodiments 1-26.

[00126] Embodiment 43 provides an aqueous emulsion comprising the hydroxylamine-termined polysiloxane of any one of Embodiments 1-26.

[00127] Embodiment 44 provides the emulsion of Embodiment 43, wherein the emulsion has an average particle size of about 0.01 microns to about 100 microns.

[00128] Embodiment 45 provides a method comprising treating at least one mammalian hair, textile, or fiber with a composition comprising the emulsion of any one of Embodiments 43-44.

[00129] Embodiment 46 provides a method of textile treatment, the method comprising: contacting a textile with a composition comprising the hydroxylamine-termined polysiloxane of any one of Embodiments 1-26.

[00130] Embodiment 47 provides a method of hair treatment, the method comprising: contacting at least one mammalian hair with a composition comprising the hydroxylamine-termined polysiloxane of any one of Embodiments 1-26.

[00131] Embodiment 48 provides the apparatus or method of any one or any combination of Embodiments 1-47 optionally configured such that all elements or options recited are available to use or select from.
CLAIMS

What is claimed is:

1. A hydroxylamine-terminated polysiloxane having the following structure:
   \[ E-R^3-[SiR^1R^2O]_DPSiR^1R^2-R^3-E \]
   wherein \( E \) is a monovalent hydroxylamino functional group,
   each of \( R^1 \) and \( R^2 \) is at each occurrence independently at least one of straight
   chain, branched, and cyclic, substituted or unsubstituted (C\(_{1-20}\))hydrocarbyl, with the
   proviso that \( R^1 \) and \( R^2 \) do not include an amino-functional group,
   \( R^3 \) is at each occurrence independently at least one of straight chain, branched,
   and cyclic, substituted or unsubstituted (C\(_{1-20}\))hydrocarbylene optionally interrupted by
   1-3 oxygen atoms, and
   DP is about 1 to about 500.

2. The polysiloxane of claim 1, wherein each of \( R^1 \) and \( R^2 \) are substituted or
   unsubstituted (C\(_{1-10}\))alkyl.

3. The polysiloxane of any one of claims 1-2, wherein \( R^3 \) is -R\(^4\)-A-R\(^5\)-, wherein
   A is -O- or a bond,
   \( R^4 \) is at least one of straight-chain, cyclic, and branched, substituted or
   unsubstituted (C\(_{2-10}\))hydrocarbylene, and
   \( R^5 \) is a bond or at least one of straight-chain, cyclic, and branched, substituted or
   unsubstituted (C\(_{1-10}\))hydrocarbylene.

4. The polysiloxane of claim 3, wherein each of \( R^4 \) and \( R^5 \) is independently (C\(_{2-}
   C_{10}\))alkylene.
5. The polysiloxane of any one of claims 1-4, wherein E is \(-Y-R^6\), wherein
    Y is a ring-opened epoxide,
    
    \(R^6\) is \(-NR^7R^8\),

    each of \(R^7\) and \(R^8\) at each occurrence is independently H or at least one of
    branched, straight-chain, and cyclic, substituted or unsubstituted \((C_1-C_{30})\)hydrocarbyl
    optionally substituted with at least one hydroxyl.

6. The polysiloxane of claim 5, wherein at least one of \(R^7\) and \(R^8\) is hydroxyl-
    substituted.

7. The polysiloxane of any one of claims 5-6, wherein Y is

8. The polysiloxane of any one of claims 5-7, wherein \(R^6\) is

\[\text{Diagram images of chemical structures}\]
9. The polysiloxane of any one of claims 1-8, wherein \(-R^3\)-E is

\[
\text{OH} \quad \text{O} \quad \text{R}^6
\]

wherein \(R^6\) is \(-NR^7R^8\),

each of \(R^7\) and \(R^8\) at each occurrence is independently at least one of straight-chain, branched, and cyclic, substituted or unsubstituted (\(C_1-C_30\)) hydrocarbyl, wherein at least one of \(R^7\) and \(R^8\) is hydroxyl-substituted.

10. The polysiloxane of any one of claims 1-9, wherein the polysiloxane has the following structure:

\[
E-R^3-[\text{SiR}^1\text{R}^2\text{O}]_{\text{DPSiR}^1\text{R}^2-R^3}\cdot\text{E}
\]

wherein \(E\) is monovalent hydroxylamino functional group \(-Y-R^6\), wherein \(Y\) is one of:

\[
\begin{align*}
\text{OH} & , \\
\text{OH} & , \\
\text{HO} & ,
\end{align*}
\]

\[
\begin{align*}
\text{HO} & , \\
\text{HO} & ,
\end{align*}
\]

\[
\text{OH} & , \text{and}
\]

\[
\text{R}^6 \quad \text{is} \quad -NR^7R^8, \text{and}
\]
each of R⁷ and R⁸ at each occurrence is independently H or at least one of straight-chain, branched, and cyclic, substituted or unsubstituted (C₁-C₃₀)hydrocarbonyl optionally substituted with at least one hydroxyl,

each of R¹ and R² is methyl or ethyl,

R³ is -R⁴-A-R⁵-, wherein

A is -O- or a bond,

R⁴ is a (C₂-C₁₀)hydrocarbylene, and

R⁵ is a bond or a substituted or unsubstituted (C₁-C₁₀)hydrocarbylene, and

DP is about 1 to about 400.

11. A composition comprising: the hydroxylamine-terminated polysiloxane of any one of claims 1-10.

12. The composition of claim 11, wherein the composition comprises a hair care composition, textile treatment composition, or aqueous emulsion.

13. A method of textile treatment, the method comprising:

contacting a textile with a composition comprising the hydroxylamine-terminated polysiloxane of any one of claims 1-10.

14. A method of hair treatment, the method comprising:

contacting at least one mammalian hair with a composition comprising the hydroxylamine-terminated polysiloxane of any one of claims 1-10.

15. A method of making a polysiloxane, the method comprising:

obtaining or providing an epoxy-terminated diorganopolysiloxane having the structure

\[ R^9.R^3-[SiR^1R^2O]_{DP}SiR^1R^2.R^3.R^9 \]

wherein R⁹ is a monovalent epoxide-containing functional group; and

reacting the epoxy-terminated diorganopolysiloxane with a primary or secondary amine, to provide a hydroxylamine-terminated polysiloxane having the following structure:

\[ E.R^3-[SiR^1R^2O]_{DP}SiR^1R^2.R^3.E \]

wherein E is a monovalent hydroxylamino functional group,
each of $R^1$ and $R^2$ is at each occurrence independently at least one of straight chain, branched, and cyclic, substituted or unsubstituted (C$_1$-C$_{20}$)hydrocarbyl, with the proviso that $R^1$ and $R^2$ do not include an amino-functional group,

$R^3$ is at each occurrence independently at least one of straight chain, branched, and cyclic, substituted or unsubstituted (C$_1$-C$_{20}$)hydrocarbylene optionally interrupted by 1-3 oxygen atoms, and

DP is about 1 to about 500.

16. A method of making a polysiloxane, the method comprising:

obtaining or providing a hydroxylamino-terminated 1,1,3,3-tetraorganodisiloxane having the following structure

$$E-R^3-SiR^1R^2O SiR^1R^2-R^3-E$$; and

reacting the hydroxylamino-terminated 1,1,3,3-tetraorganodisiloxane with a cyclopolydiorganosiloxane, to provide a hydroxylamine-terminated polysiloxane having the following structure:

$$E-R^3-[SiR^1R^2O]_{DP}SiR^1R^2-R^3-E$$

wherein $E$ is a monovalent hydroxylamino functional group,

each of $R^1$ and $R^2$ is at each occurrence independently at least one of straight chain, branched, and cyclic, substituted or unsubstituted (C$_1$-C$_{20}$)hydrocarbyl, with the proviso that $R^1$ and $R^2$ do not include an amino-functional group,

$R^3$ is at each occurrence independently at least one of straight chain, branched, and cyclic, substituted or unsubstituted (C$_1$-C$_{20}$)hydrocarbylene optionally interrupted by 1-3 oxygen atoms, and

DP is about 1 to about 500.
INTERNATIONAL SEARCH REPORT

International application No
PCT/US2014/014085

A. CLASSIFICATION OF SUBJECT MATTER

INV. C08G77/10 C08G77/26 C08G77/388 C08L83/08 A61K8/89

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C08G C08L A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
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<td>5-10, 15, 16</td>
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Further documents are listed in the continuation of Box C. X See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance
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<table>
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