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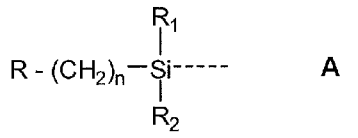
(54) Title: IMIDOALKYL SILOXANE NANOCOMPOSITES



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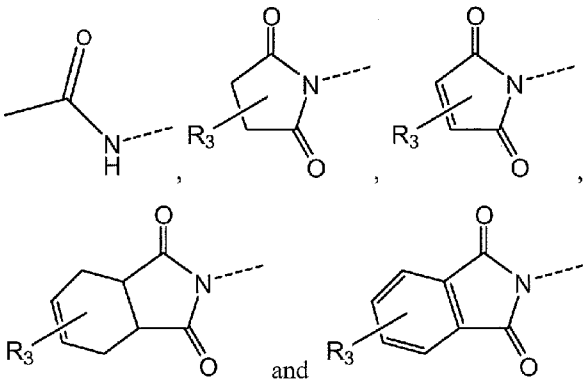

B

(57) Abstract: The present invention relates to a composition comprising a silica particle wherein silanol groups on the surface of the silica particle are functionalized with an imidoalkyl siloxane of formula A (see A) where n is selected from 1, 2, 3, 4, 5, 6, 7 and 8, where R_1 is selected from hydrogen, alkyl and a hydrolysable group, where R_2 is selected from hydrogen, alkyl and a hydrolysable group, where R is selected from (see compositions B) where R_3 is selected from hydrogen, alkyl, aryl, alkoxy, hydroxy, amide, amine, ester and -COOH; and wherein the silica particle functionalized with an imidoalkyl siloxane has a particle size of about 100 nanometers or less. The present invention also includes articles comprising such compositions and processes to produce such compositions.

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IMIDOALKYL SILOXANE NANOCOMPOSITES

CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] This application claims the benefit of the filing date of U.S. provisional application No. 61/036,651, filed on March 14, 2008, the entire contents of which are incorporated herein by reference.

FIELD OF THE INVENTION

[0002] The present invention relates to imidoalkyl siloxane functionalized silica particles useful in polymer compositions.

BACKGROUND OF THE INVENTION

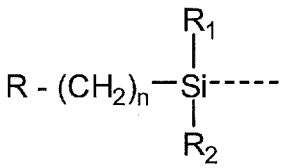
[0003] Over the past decade the focus of research activities related to reinforced polymeric composites is in the field of polymer nanocomposites. Small quantities of fillers that have at least one dimension in the nanometer range have provided some improvements in mechanical properties. Silica nanoparticles have received considerable attention owing to their 3-dimensional nanoscale and reactive surface due to the presence of silanol groups. The homogeneous dispersion of silica nanoparticles in a polymer matrix was a key problem due to the intrinsic agglomeration tendency of the nanoparticles. This problem was solved by treating the silica surface with various organic substances.

[0004] The surface of silica (SiO_2) can be functionalized with various organic moieties, by reaction of the silanol groups on the silica surface. Reactive siloxanes such as chloro–alkoxy silanes and many different silanes with a large variety of organic moieties are commercially available. The most often used for functionalization is 3-aminopropyl siloxane. This primary amine is a good ligand for metal ions and can therefore serve as sorbent in waste-water treatment, or can immobilize catalytically active transition metal ions. Furthermore, the nucleophilic primary amine can be used as linker between silica surface and any organic species.

[0005] Unfortunately, these materials have poor hydrothermal stability in humid environment or aqueous solutions. Additionally, the amine groups have a tendency to form carbamic acids in the presence of atmospheric CO_2. Finally, the use of nanosilica to reinforce polyester composites is limited by the poor thermal stability of the functionalized silica nanoparticles leading to decomposition during polymerization and extrusion. Therefore, a need exists for functionalized nanosilica particles with higher temperature stability than current compositions.

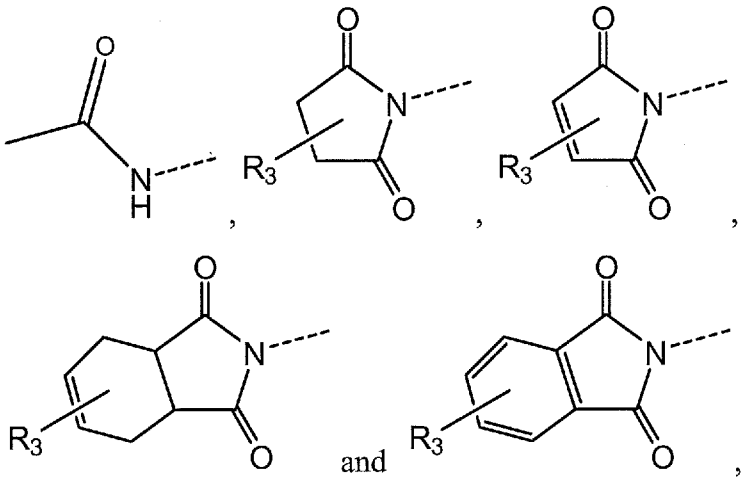
SUMMARY OF THE INVENTION

[0006] In accordance with the present invention, it has now been found that functionalizing silica particles with imidoalkyl siloxanes improve temperature stability as compared to currently known functionalized silica particles. The present invention relates to a composition comprising a silica particle wherein silanol groups on the surface of the silica particle are functionalized with an imidoalkyl siloxane of formula A,



A

where n is selected from the group consisting of 1, 2, 3, 4, 5, 6, 7 and 8,
where R_1 is selected from the group consisting of hydrogen, alkyl and a hydrolysable group,
where R_2 is selected from the group consisting of hydrogen, alkyl and a hydrolysable group,
where R is selected from the group consisting of



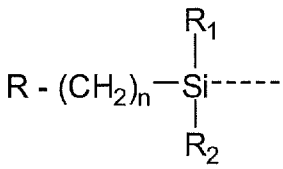
where R_3 is selected from the group consisting of hydrogen, alkyl, aryl, alkoxy, hydroxy, amide, amine, ester and –COOH;

and

wherein the silica particle functionalized with an imidoalkyl siloxane has a particle size of about 100 nanometers or less. The present invention also includes articles comprising such compositions and processes to produce such compositions.

DETAILED DESCRIPTION OF THE INVENTION

[0007] The present invention can be characterized by a composition comprising a silica particle wherein silanol groups on the surface of the silica particle are functionalized with an imidoalkyl siloxane of formula A,



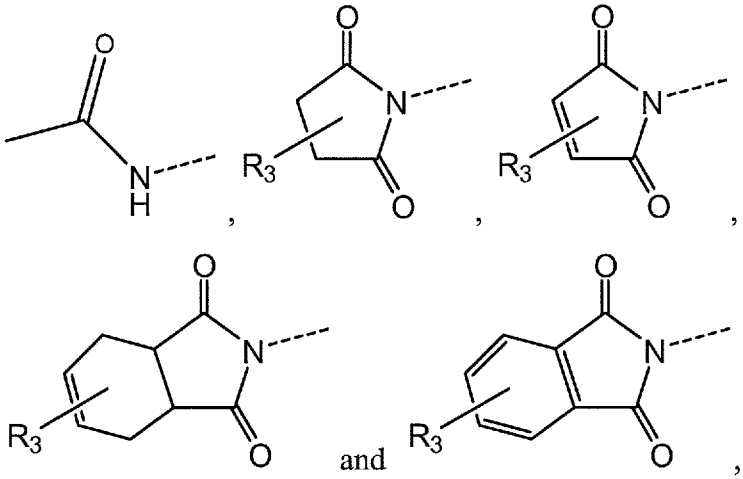
A

where n is selected from the group consisting of 1, 2, 3, 4, 5, 6, 7 and 8,

where R_1 is selected from the group consisting of hydrogen, alkyl and a hydrolysable group,

where R_2 is selected from the group consisting of hydrogen, alkyl and a hydrolysable group,

where R is selected from the group consisting of



, where R_3 is selected from the group consisting of hydrogen, alkyl, aryl, alkoxy, hydroxy, amide, amine, ester and –COOH;

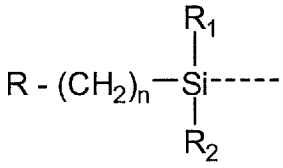
and

wherein the silica particle functionalized with an imidoalkyl siloxane has a particle size of about 100 nanometers or less. The dotted lines in the formulae above represent bond location to the connected molecule. The molar mass of imidoalkyl siloxane on the surface of the silica can be greater than about 1 mmol g^{-1}, for example greater than about 1.2 mmol g^{-1} or greater than about 1.4 mmol g^{-1}. In formula A above, n can be 2, 3, 4, 5 or 6, for example 2, 3 or 4, or n can be 3; R_1 can be, for example, methyl or hydroxyl; and R_2 can be, for example, methyl or hydroxyl. The functionalized silica particle size can be about 75 nanometers or less, for example 50 nanometers or less or 20 nanometers or less.

[0008] The composition of the present invention can further comprise a polyester. The polyester can be selected from the group consisting of polyethylene terephthalate, polybutylene terephthalate, polypropylene terephthalate, poly (1,4 cyclohexylene-dimethylene) terephthalate, polyethylene naphthalate, polyethylene bibenzoate, and copolyesters of these. The polyester can be present in a concentration of from about 80 weight % to about 99.9 weight %, for example from about 90 weight % to about 99.9 weight % or from about 95 weight % to about 99.9 weight %. The concentration of the imidoalkyl siloxane functionalized silica particles in the polyester can be in the range of from about 0.5 to about 10% by weight, for example in the range of from about 1 about 5% by weight, based on the weight of the polyester.

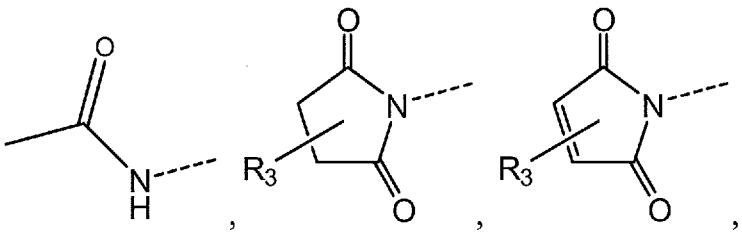
[0009] The present invention also includes articles made from the above compositions. For example, articles can be sheets, films, fibers or other injection molded articles such as containers.

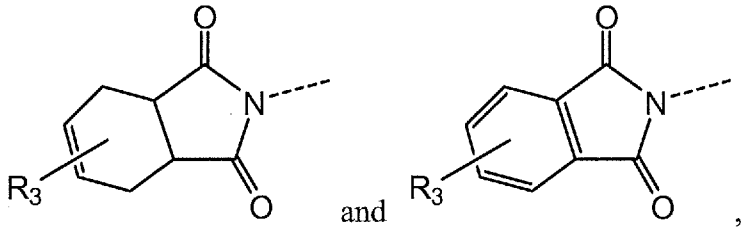
[0010] Another embodiment of the present invention is a process for preparing an imidoalkyl siloxane functionalized silica particle comprising reacting an amino-functionalized silica particle with a carboxylic acid anhydride to form a silica particle functionalized with an imidoalkyl siloxane of formula A,



A

where n is selected from the group consisting of 1, 2, 3, 4, 5, 6, 7 and 8,
where R_1 is selected from the group consisting of hydrogen, alkyl and a hydrolysable group,
where R_2 is selected from the group consisting of hydrogen, alkyl and a hydrolysable group,
where R is selected from the group consisting of





and

, where R_3 is selected from the group consisting of hydrogen, alkyl, aryl, alkoxy, hydroxy, amide, amine, ester and –COOH.

[0011] Any carboxylic acid anhydride can be used to react with the amino-functionalized silica particle. Examples of these anhydrides are aliphatic anhydrides

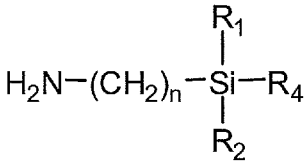
such as acetic anhydride; cyclic anhydrides such as maleic, glutaric, phthalic and diphenic anhydrides; and substituted cyclic anhydrides, where the substituents can be short/long chain alkyl, aryl, alkoxy/hydroxy, amides/amines, ester or carboxylic, such as methyl succinic, phenyl succinic methyl maleic, dimethyl maleic, methyl phthalic and tetrahydrophthalic anhydride. Suitable carboxylic acid anhydrides can be acetic, succinic, maleic, phthalic and tetrahydrophthalic anhydride. A single anhydride or a mixture of anhydrides can be used.

[0012] The amino-functionalized silica particles can be treated with the carboxylic acid anhydride in an organic solvent, for example toluene, to produce a dry powder, or *in-situ* polymerization of polyester, in the glycol. The molar ratio of the anhydride to amine group of the amino-functionalized silica can be in the range of about 0.8 to 1.5, for example in the range of about 0.9 to 1.3 or about 1:1.

[0013] In the above process, the molar mass of imidoalkyl siloxane on the surface of the silica can be greater than about 1 mmol g^{-1}, for example greater than about 1.2 mmol g^{-1} or greater than about 1.4 mmol g^{-1}.

[0014] Another embodiment of the present invention is a process for preparing a functionalized silica particle comprising:

a) reacting silica particles with an amino-silane of formula B

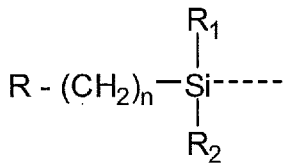


B

where n is selected from the group consisting of 1, 2, 3, 4, 5, 6, 7 and 8,
where R_1 is selected from the group consisting of hydrogen, alkyl and a hydrolysable group,
where R_2 is selected from the group consisting of hydrogen, alkyl and a hydrolysable group,
where R_4 is a hydrolysable group,

in an organic solvent to form an amino-functionalized silica particle; and

b) reacting the amino-functionalized silica particle with a carboxylic acid anhydride to form a silica particle functionalized with an imidoalkyl siloxane of formula A,



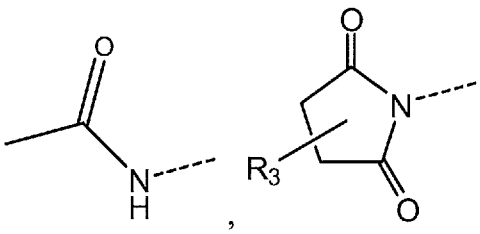
A

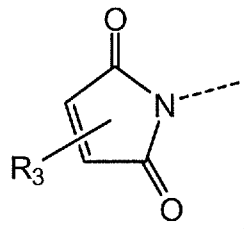
where n is as specified above,

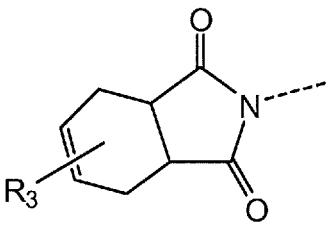
where R_1 is as specified above,

where R_2 is as specified above,

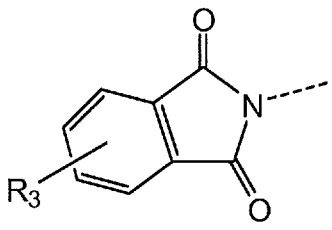
where R is selected from the group consisting of







and



, where R_3 is selected from the group consisting of hydrogen, alkyl, aryl, alkoxy, hydroxy, amide, amine, ester and –COOH.

[0015] The silica particles can be reacted with the amino-silane in an organic solvent, for example toluene, xylene, dimethyl sulfoxide, N,N-dimethyl formamide or other alcohols, to produce a dry powder, or for in-situ polymerization of polyester, in the glycol. The molar ratio of the silane to the hydroxyl content of the silica can be in the range of about 0.25 to 1.5, for example in the range of about 0.75 to 1.25 or about 1:1. Process conditions are chosen so that the molar mass of imidoalkyl siloxane on the surface of the silica is greater than about 1 mmol g^{-1}, for example greater than about 1.2 mmol g^{-1} or greater than about 1.4 mmol g^{-1}.

[0016] The hydrolysable group of R_1, R_2 or R_4 can be methoxy, ethoxy or acetoxy. For example, the amino-silane of formula **B** can be 3-aminopropyl-triethoxysilane, 3-aminopropyl-trimethoxy-silane, 3-aminoethyl-triethoxysilane, 3-aminobutyl-triethoxysilane, 3-aminoethyl-trimethoxysilane or 3-amino-propyl-methyl-diethoxysilane. A single amino-silane or a mixture of amino-silanes can be used.

[0017] The amino-functionalized silica particles can be reacted with a carboxylic acid anhydride in a solvent, for example toluene, to produce a dry powder, or *in-situ* polymerization of polyester, in the glycol. The molar ratio of the anhydride to amine group of the amino-functionalized silica can be in the range of about 0.8 to 1.5, for example in the range of about 0.9 to 1.3 or about 1:1.

[0018] Any carboxylic acid anhydride can be used to react with the amino-functionalized silica particle. Examples of these anhydrides are aliphatic anhydrides such as acetic anhydride; cyclic anhydrides such as maleic, glutaric, phthalic and diphenic anhydrides; and substituted cyclic anhydrides, where the substituents can be short/long chain alkyl, aryl, alkoxy/hydroxy, amides/amines, ester or carboxylic, such as methyl succinic, phenyl succinic methyl maleic, dimethyl maleic, methyl phthalic and tetrahydrophthalic anhydride. Suitable carboxylic acid anhydrides can be acetic, succinic, maleic, phthalic and tetrahydrophthalic anhydride. A single anhydride or a mixture of anhydrides can be used.

[0019] The reacting of the silica particles with the amino-silane and the subsequent reacting with a carboxylic acid anhydride to form the imidoalkyl siloxane functionalized silica particle can be performed in a common solvent without isolating the functionalized product. For example, the silica particles can be dispersed in the glycol in a heated stirred reactor, the amino-silane added and reacted and then the carboxylic acid anhydride can be added to the slurry and reacted. The temperature of the glycol can be within 50° C of its boiling point, or refluxed at its boiling point. The reaction time for each reaction can be about 7-8 hours. The slurry can be cooled for subsequent addition with the aromatic acid and glycol to prepare the polyester. The concentration of the functionalized silica particles in the slurry can be in the range of

from about 3 to about 30% by weight, for example in the range of from about 5 about 20% by weight, based on the weight of the slurry.

[0020] Generally, the polyester can be produced by conventional polymerization process from an aromatic dicarboxylic acid or an ester-forming derivative and glycol as starting materials. Examples of the aromatic dicarboxylic acid used in the present invention include terephthalic acid, isophthalic acid, 2,6-naphthalenedicarboxylic acid, phthalic acid, adipic acid, sebacic acid and mixtures thereof. The aromatic acid moiety can be at least 85 mole % of terephthalic acid. Examples of the glycol that can be used in the present invention include ethylene glycol, butanediol, propylene glycol, and 1,4-cyclohexanedimethanol, and mixtures thereof. The primary glycol can be at least 85 mole % of ethylene glycol, butanediol, propylene glycol or 1,4-cyclohexanedimethanol.

[0021] Conventional polyester processes can be used in this invention, using either transesterification of the ester derivative of the aromatic acid, or direct esterification of the aromatic acid with the glycol. The imidoalkyl siloxane functionalized silica particle slurry being added during this first stage. The polymerization can be catalyzed by conventional catalysts such as antimony, titanium, germanium, aluminum compounds. After polymerization the polyester can be extrude into strands, quench with water, cut into pellets, dried and crystallized. For high molecular weight polyesters, these resin pellets can be solid state polymerized by conventional methods.

[0022] The addition of a variety of conventionally known additives is also within the scope of the present invention. Accordingly, heat stabilizers, anti-blocking agents, antioxidants, antistatic agents, UV absorbers, various pigments, dyes, fillers, branching agents, and other typical agents can be added to the polymer generally during or near the end of the polycondensation reaction. Conventional systems can be employed for the introduction of additives to achieve the desired result.

[0023] As used in this specification and unless otherwise indicated the term "alkyl" used alone or as part of a larger moiety, includes straight or branched chains

of at least one or two carbon atoms, as appropriate to the substituent, and up to 18 carbon atoms, for example up to ten carbon atoms or up to seven carbon atoms.

Test Methods

[0024] Thermogravimetric analyses (TGA) were performed on a TA Instruments SDT 2960 simultaneous DTA-TGA, at a rate of 10 °C/minute up to 700 °C under nitrogen atmosphere.

[0025] The molar mass of the imidoalkyl siloxane on the surface of the silica particle is calculated from the weight loss between 200° and 600° C. After the loss of mass due to the physisorbed water from the silica surface (< 150° C), there is a gradual loss associated with the removal of the organic content of the imidoalkyl siloxane. Between 200° - 600° C the organic content of the imidoalkyl siloxane is removed and any silanol groups of the imidoalkyl siloxane condense to form siloxane groups with the loss of water. It is assumed that there is no mass loss due to the condensation of free silanol groups on the surface of the silica.

[0026] If the molar mass of the imidoalkyl siloxane is M, the molar mass of the material removed is (M – Si – O) or M*, i.e. only a siloxane group remains. If neither R_1 nor R_2 in formula A are hydrolysable groups, M* = M – Si. The molar mass of the imidoalkyl siloxane on the surface of the silica particle is calculated from the following equations:

[0027] Molar mass lost (M*)/ gram of silica = [(% weight loss/100) / M*] = W

[0028] Molar mass of imidoalkyl siloxane/gram of silica (mol g^{-1}) = M x W/M*

[0029] The surface hydroxyl content of the silica particle is also calculated from the mass loss of the silica between 200° and 600° C. The condensation of the silanol groups on the surface occurs by the reaction of two silanol groups to form one siloxane group with the release of water. For a weight loss of W_1 percent, the molar hydroxyl content per gram of silica is calculated from the following equation:

[0030] Molar mass of hydroxyl groups/gram of silica (mol g^{-1}) = 2 x W_1/(18 x 100)

[0031] Solid-state NMR spectroscopy was performed on a Varian INOVA 400 MHz spectrometer using a 3 channel HXY 4 mm Chemagnetic probe. Samples were loaded into loaded into zirconia sleeves with Teflon caps and spun at 7 to 9 kHz during acquisition to identify spinning sidebands. The acquisition parameters were as follows: ^{13}C 90° pulse width was 3.5 μs, acquisition time was 45 ms, and recycle delay time was 4 s.

[0032] Dynamic Mechanical Analysis (DMA) measurements were performed on the samples under nitrogen using a TA Instrument at a heating rate of 2° C/minute at a frequency of 1 HZ and a force of 0.1 N.

[0033] Intrinsic Viscosity (IV) of the polymer was measured according to ASTM D4603-96.

Examples

Comparative Example 1

[0034] 50 g of silica nanoparticles (Degussa Aerosil®200, particle size of 12 nm) were dispersed in 1L of toluene under nitrogen at 80 °C for 30 min. To this dispersion, 50 g of 3-aminopropyltrimethoxy silane (APS) (Gelest, Inc) in 20 ml toluene was added instantaneously. The mixture was refluxed for 18 h. The product was collected via vacuum filtration on a Buchner funnel, washed several times with methanol and acetone successively to remove unreacted APS, and dried in a vacuum oven at 80 °C for 24 h. This compound comprised 1.6 mmol g^{-1} of the amino siloxane derivative, corresponding to 1.9 mmol of NH_2 groups/g silica

Example 2

[0035] 5 g of the APS-treated silica product of Example 1 and various anhydrides (9.5 mmol) were added to a 500-mL round-bottomed flask purged with nitrogen. The mixture was stirred and refluxed in 150 mL of toluene for 24 h. The product was collected via vacuum filtration on a Buchner funnel, washed several

times with methanol and acetone successively, and dried in a vacuum oven at 80 °C for 24 h.

[0036] The resultant imidoalkyl siloxanes were analyzed by TGA, and the results are summarized in terms of peak decomposition temperatures, percent weight loss (200 - 600 °C) and molar mass of the imidoalkyl siloxane on the silica in Table 1.

Table 1

Reactants	**Peak decomposition temperature (° C)**	**Weight loss (200°-600° C), %**	**Imidoalkyl siloxane, mmol g^{-1}**
SiO_2/APS	387	11.8	--
SiO_2/APS/Acetic anhydride	414	16.9	1.6
SiO_2/APS/Succinnic anhydride	490	18.5	1.2
SiO_2/APS/Maleic anhydride	513	21.7	1.4
SiO_2/APS/Phthalic anhydride	515	31.8	1.6
SiO_2/APS/Tetrahydrophthalic anhydride	493	32.7	1.6

[0037] It was observed that the thermal stability of the SiO_2/amide or imide was much higher than precursor SiO_2/APS.

[0038] The molar mass of hydroxyl groups on the surface of silica particles is reported to be in the range of 1.4 to 1.9 mmol g^{-1}. The molar mass of the imidoalkyl siloxane functionalized silica reported in table 1 is in the range of surface hydroxyl groups initially present on silica. The surface hydroxyl groups have therefore been functionalized to a high extent.

[0039] This high degree of functionalization is also supported by the ^{13}C solid-state NMR spectra obtained for these samples. The ^{13}C spectra obtained for the APS/SiO_2 material of Comparative Example 1 shows three peaks at 42, 22.5 and 9.9 ppm corresponding to the methylene groups of APS, i.e. N-CH_3, Si-CH_3 and –CH_2 respectively. The carbon attached to nitrogen is more downfield as compared to the carbon attached to silicon, while the methylene group in the middle is most upfield as expected. The products obtained from further functionalization of SiO_2/APS to form imidoalkyl siloxanes (Example 2) were also characterized by solid-state ^{13}C NMR spectroscopy. All the spectra obtained for these materials show three characteristic peaks corresponding to the methylene groups of the APS in region between 10-50 ppm. In addition a new peak between 170-180 ppm for all the functionalized samples was observed, which is consistent with the presence carbonyl group in these materials. Thus corroborates the formation of imidoalkyl siloxane nanoparticles.

Comparative Example 3

[0040] 100 g ethylene glycol was heated with rapid stirring to 120°C and 100 g of silica nanoparticles (Degussa Aerosil®200, particle size of 12 nm) was added, and held at 120°C for 1 hour with stirring. 26.5 g of APS was added and stirred at 120°C for 8 hours, then cooled.

Example 4

[0041] 100 g ethylene glycol was heated with rapid stirring to 120°C and 100 g of silica nanoparticles (Degussa Aerosil®200, particle size of 12 nm) was added, and held at 120°C for 1 hour with stirring. 26.5 g of APS was added and stirred at 120°C for 8 hours. 7.2 g acetic anhydride (AcOAc) was added to the slurry and stirred at 120° C for 8 hours, and the dispersion cooled.

Example 5

[0042] Polyester (PET) resins were prepared by a standard polymerization process using terephthalic acid, ethylene glycol and an antimony catalyst. Slurries of Examples 3 and 4 were added to give a wt.-% of silica of 1 % based on the polymer. The amorphous polymer pellets had a nominal IV of 0.6. They were solid stated polymerized to a nominal IV of 0.8.

[0043] These resins, together with a control PET resin without the silica compounds, were injection molded into preforms and stretch blow molded into 500 ml bottles. Sections from the bottle sidewalls (nominally 0.3 mm thick), in the axial direction, were cut and the storage modulus (DMA) measured, the results are set forth in Table 2.

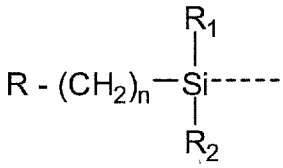
Table 2.

Sample composition	Storage modulus, MPa		
	30° C	150° C	200° C
PET control	3340	346	199
Comparative Example 3	3170	460	335
Example 4	4821	708	446

[0044] Thus it is apparent that there has been provided, in accordance with the invention, an imidoalkyl siloxane functionalized silica nanoparticle that fully satisfies the aims, advantages and objects set forth above. While the invention has been described in conjunction with specific embodiments thereof, it is evident that the many alternatives, modifications, and variations will be apparent to those skilled in the art in light of the foregoing description. Accordingly, the invention is intended to embrace all such alternatives, modifications and variations as fall within the spirit and scope of the claims.

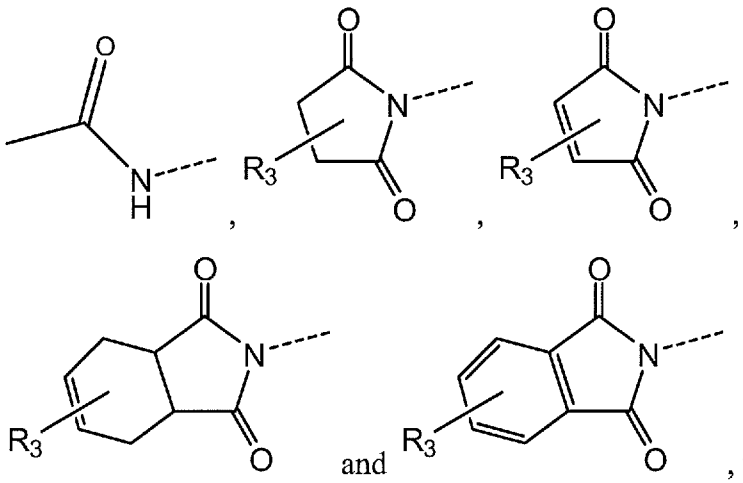
What is claimed is:

1) A composition comprising a silica particle wherein silanol groups on the surface of the silica particle are functionalized with an imidoalkyl siloxane of formula A,



A

where n is selected from the group consisting of 1, 2, 3, 4, 5, 6, 7 and 8,
where R1 is selected from the group consisting of hydrogen, alkyl and a hydrolysable group,
where R_2 is selected from the group consisting of hydrogen, alkyl and a hydrolysable group,
where R is selected from the group consisting of



and , where R_3 is selected from the group consisting of hydrogen, alkyl, aryl, alkoxy, hydroxy, amide, amine, ester and –COOH;
and
wherein the silica particle functionalized with an imidoalkyl siloxane has a particle size of about 100 nanometers or less.

2) The composition of claim 1 wherein the imidoalkyl siloxane is present at a molar mass of greater than about 1 mmol g^{-1} of said silica particle.

3) The composition of claim 1 wherein the imidoalkyl siloxane is present at a molar mass of greater than about 1.2 mmol g^{-1} of said silica particle

4) The composition of claim 1 wherein n is selected from the group consisting of 2, 3, 4, 5 and 6.

5) The composition of claim 1 wherein n is selected from the group consisting of 2, 3 and 4.

6) The composition of claim 1 wherein n is 3.

7) The composition of claim 1 wherein R_1 is methyl or hydroxyl.

8) The composition of claim 1 wherein R_1 is hydroxyl.

9) The composition of claim 1 wherein R_2 is methyl or hydroxyl.

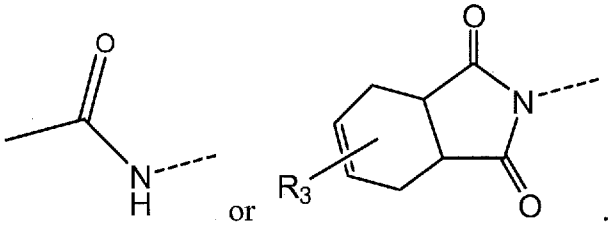
10) The composition of claim 1 wherein R_2 is hydroxyl.

11) The composition of claim 1 wherein the particle size is about 75 nanometers or less.

12) The composition of claim 1 wherein the particle size is about 50 nanometers or less.

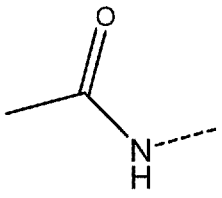
13) The composition of claim 1 wherein the particle size is about 20 nanometers or less.

14) The composition of claim 1 wherein R is



15) The composition of claim 1 wherein R_3 is hydrogen or alkyl.

16) The composition of claim 1 wherein n is 3, R is



, R_1 is hydroxyl, and R_2 is hydroxyl.

17) The composition of claim 1 further comprising a polyester.

18) The composition of claim 17 wherein said polyester is selected from the group consisting of polyethylene terephthalate, polybutylene terephthalate, polypropylene terephthalate, poly(1,4 cyclohexylene-dimethylene) terephthalate, polyethylene naphthalate, polyethylene bibenzoate, and copolyesters of these.

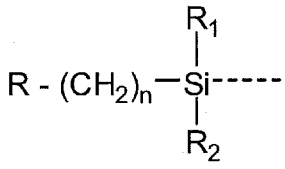
19) The composition of claim 17 wherein the polyester is present in a concentration of from about 80 weight % to about 99.9 weight %.

20) The composition of claim 17 wherein the polyester is present in a concentration of from about 90 weight % to about 99.9 weight %.

21) The composition of claim 17 wherein the polyester is present in a concentration of from about 95 weight % to about 99.9 weight %.

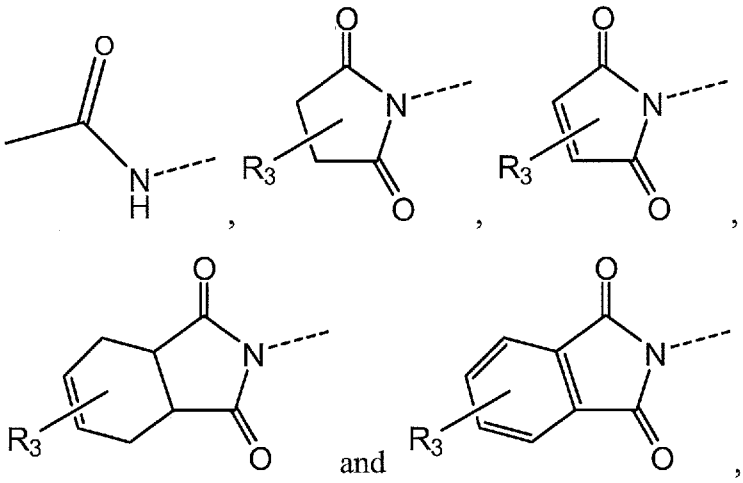
22) An article comprising the composition of either claim 1 or 17.

23) A process for preparing a imidoalkyl siloxane functionalized silica particle comprising reacting an amino-siloxane functionalized silica particle with a carboxylic acid anhydride to form a silica particle functionalized with an imidoalkyl siloxane of formula A,



A

where n is selected from the group consisting of 1, 2, 3, 4, 5, 6, 7 and 8,
where R_1 is selected from the group consisting of hydrogen, alkyl and a hydrolysable group,
where R_2 is selected from the group consisting of hydrogen, alkyl and a hydrolysable group,
where R is selected from the group consisting of



and

, where R_3 is selected from the group consisting of hydrogen, alkyl, aryl, alkoxy, hydroxy, amide, amine, ester and –COOH.

24) The process of claim 23 wherein the carboxylic acid anhydride is selected from the group consisting of aliphatic anhydrides, unsubstituted cyclic anhydrides and substituted cyclic anhydrides where the substituent is selected from the group consisting of alkyl, aryl, alkoxy, hydroxyl, amides, amines, ester and carboxylic.

25) The process of claim 24 wherein the carboxylic acid anhydride is selected from the group consisting of acetic anhydride, maleic anhydride, glutaric anhydride, phthalic anhydride, diphenic anhydride, methyl succinic anhydride, phenyl succinic anhydride, methyl maleic anhydride, dimethyl maleic anhydride, methyl phthalic anhydride and tetrahydrophthalic anhydride.

26) The process of claim 23 wherein the reacting is performed in an organic solvent.

27) The process of claim 26 wherein the organic solvent is toluene.

28) The process of claim 23 wherein the reacting is performed in glycol during in-situ polymerization of a polyester.

29) The process of claim 23 wherein the carboxylic acid anhydride is present at a molar ratio of carboxylic acid anhydride to amine group of the amino-functionalized silica particle in the range of from about 0.8 to about 1.5.

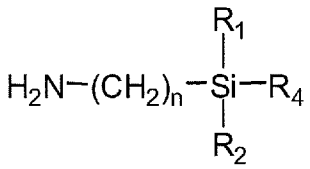
30) The process of claim 23 wherein the imidoalkyl siloxane is present at a molar mass of greater than about 1 mmol g^{-1} of said silica particle.

31) The process of claim 23 wherein the imidoalkyl siloxane is present at a molar mass of greater than about 1.2 mmol g^{-1} of said silica particle.

32) The process of claim 23 wherein the imidoalkyl siloxane is present at a molar mass of greater than about 1.4 mmol g^{-1} of said silica particle.

33) A process for preparing a functionalized silica particle comprising:

a) reacting silica particles with an amino-silane of formula B



B

where n is selected from the group consisting of 1, 2, 3, 4, 5, 6, 7 and 8,

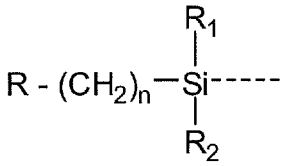
where R_1 is selected from the group consisting of hydrogen, alkyl and a hydrolysable group,

where R_2 is selected from the group consisting of hydrogen, alkyl and a hydrolysable group,

where R_3 is a hydrolysable group,

in an organic solvent to form an amino-functionalized silica particle; and

b) reacting the amino-functionalized silica particle with a carboxylic acid anhydride to form a silica particle functionalized with an imidoalkyl siloxane of formula A,



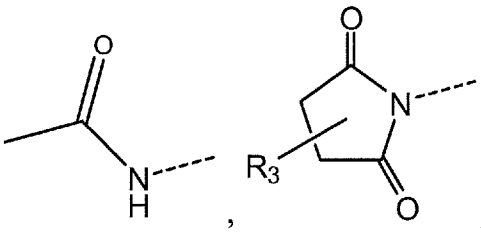
A

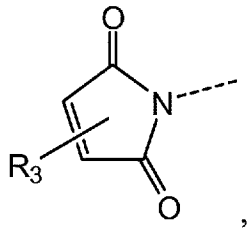
where n is as specified above,

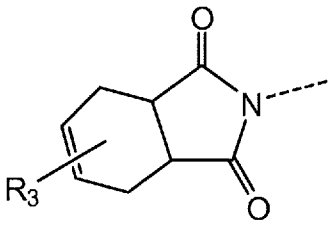
where R_1 is as specified above,

where R_2 is as specified above,

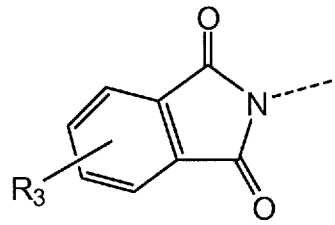
where R is selected from the group consisting of







and



, where R_3 is selected from the group consisting of hydrogen, alkyl, aryl, alkoxy, hydroxy, amide, amine, ester and –COOH.

34) The process of claim 33 wherein the carboxylic acid anhydride is selected from the group consisting of aliphatic anhydrides, unsubstituted cyclic anhydrides and substituted cyclic anhydrides where the substituent is selected from the group consisting of alkyl, aryl, alkoxy, hydroxyl, amides, amines, ester and carboxylic.

35) The process of claim 34 wherein the carboxylic acid anhydride is selected from the group consisting of acetic anhydride, maleic anhydride, glutaric anhydride, phthalic anhydride, diphenic anhydride, methyl succinic anhydride, phenyl succinic anhydride, methyl maleic anhydride, dimethyl maleic anhydride, methyl phthalic anhydride and tetrahydrophthalic anhydride.

36) The process of claim 33 wherein the reacting of step a) is performed in an organic solvent.

37) The process of claim 36 wherein the organic solvent is selected from the group consisting of toluene, xylene, dimethyl sulfoxide, N,N-dimethyl formamide and alcohol.

38) The process of claim 33 wherein the reacting of step a) is performed in glycol during in-situ polymerization of a polyester.

39) The process of claim 33 wherein the reacting of step b) is performed in an organic solvent.

40) The process of claim 39 wherein the organic solvent is toluene.

41) The process of claim 33 wherein the reacting of step b) is performed in glycol during in-situ polymerization of a polyester.

42) The process of claim 33 wherein the amino-silane is present at a molar ratio of amino-silane to hydroxyl content of the silica in the range of about 0.25 to 1.5.

43) The process of claim 33 wherein the carboxylic acid anhydride is present at a molar ratio of carboxylic acid anhydride to amine group of the amino-siloxane functionalized silica particle in the range of from about 0.8 to about 1.5.

44) The process of claim 33 wherein the imidoalkyl siloxane is present at a molar mass of greater than about 1 mmol g^{-1} of said silica particle.

45) The process of claim 33 wherein the imidoalkyl siloxane is present at a molar mass of greater than about 1.2 mmol g^{-1} of said silica particle.

46) The process of claim 33 wherein the imidoalkyl siloxane is present at a molar mass of greater than about 1.4 mmol g^{-1} of said silica particle.

47) A composition comprising a silica particle and a polyester, wherein silanol groups on the surface of the silica particle are functionalized with 3-aminopropyltrimethoxy silane.