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54 **Treating composition comprising organopolysiloxane containing polyoxyalkylene and alkoxysilylalkyl radicals.**

57 A composition for treating a solid material to give it durable hydrophilic and/or antistatic properties comprises a siloxane compound which has one or more terminal alkoxysilylalkyl groups and one or more polyoxyalkylene groups. In a preferred embodiment the composition is useful to treat fibers and fiber-containing materials. The composition can further contain a curing agent for the silicone.

EP 0 255 205 A2

TREATING AGENT COMPRISING ORGANOPOLYSILOXANE CONTAINING POLYOXYALKYLENE AND ALKOXYSILYLALKYL RADICALS

The present invention relates to an agent for treating solids. More specifically, the present invention relates to a treatment agent which imparts a durable hydrophilicity and antistaticity to solid materials.

In order to impart hydrophilicity and antistaticity to solids such as, for example, moldings, sheet-form materials, foams, fibers, and powders, treatments have heretofore been carried out using various organic surfactants, for example, cationic types, anionic types, and nonionic types, etc.

Furthermore, organopolysiloxane-polyoxyalkylene copolymer, such as that disclosed in Japanese Patent Publication Number 44-6069 (6,069/69), and organosilyl-terminated polyoxyalkylene-modified and alkoxysilylalkyl-modified organopolysiloxane, such as that disclosed in Japanese Patent Application Laid Open Number 57-139123 (139,123/82), are known as silicone-type agents for the treatment of solids.

However, treatment methods which employ organic surfactant and the treatment method using the organopolysiloxane-polyoxyalkylene copolymer as described in Japanese Patent Publication Number 44-6069 suffer from the problem of providing only a temporary hydrophilicity and antistaticity, and these effects are readily lost upon exposure to water or organic solvent.

While the silicone-type surfactant described in Japanese Patent Application Laid Open Number 57-139123 has the alkoxysilyl alkyl group as a side chain, an agent for treating solids whose principal agent is organopolysiloxane having at least 1 polyoxyalkylene group as a side chain or molecular terminal and having the alkoxysilylalkyl group at least at one molecular terminal, is unknown.

The object of the present invention is to eliminate the problems described above by providing a novel agent for treating solids which can impart a durable hydrophilicity and antistaticity to solids. It is a particular object of this invention to provide a method for conferring durable hydrophilicity and antistaticity properties to fibers and fiber-containing materials.

These objects, and others which will become apparent upon consideration of the following disclosure and appended claims, are obtained by the method of this invention which, briefly stated, comprises treating a solid material with a composition which comprises, as its principal component, an organopolysiloxane compound which contains at least one siloxane chain-terminating siloxane unit bearing an alkoxysilylalkyl radical and at least one siloxane unit bearing a polyoxyalkylene radical. In a preferred embodiment of this invention both of the siloxane chain-terminating siloxane units bear alkoxysilylalkyl radical.

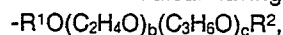
The present invention relates to a composition for treating solids, said composition comprising an organopolysiloxane compound which has the formula

$A(R_2SiO)_x(RQSiO)_y(RGSiO)_zSiR_2A$, wherein

Q denotes a radical having the formula



G denotes a radical having the formula



A denotes a radical selected from the group consisting of Q and G radicals, at least one A radical being a Q radical,

x has a value of from 5 to 500,

y has a value of from 0 to 100,

z has a value of from 0 to 100,

X denotes an alkoxy or alkoxyalkoxy radical having from 1 to 4 carbon atoms,

R denotes a monovalent hydrocarbon or halogenated hydrocarbon radical having from 1 to 10 carbon atoms,

R¹ denotes an alkylene radical having from 2 to 5 carbon atoms,

R² denotes a hydrogen atom or a monovalent organic radical having from 1 to 5 carbon atoms,

a has a value of 2 or 3,

b has a value of from 0 to 50,

c has a value of from 0 to 50, and

b plus c has a value of from 2 to 100;

there being present in said organopolysiloxane at least one G radical.

By way of explanation of the preceding, R in the above formulas is a monovalent hydrocarbon group having 1 to 10 carbon atoms, and it is exemplified by alkyl groups such as methyl, ethyl, propyl, and octyl; by substituted alkyl groups such as 3,3,3-trifluoropropyl, 2-phenylethyl and 2-phenylpropyl; by aryl groups such as phenyl and tolyl; and by substituted aryl groups. Alkyl groups, most preferably methyl, are preferred here. The groups R in a single molecule may be identical or different.

A can be a Q or G group, delineated below, with the proviso that at least one A is a Q group. Preferably all A groups are Q groups.

Q is a group having the formula $-R^1SiX_aR^{(3-a)}$ and it functions to impart durability by tightly bonding the present organopolysiloxane to solids. R^1 is to be an alkylene group having 2 to 5 carbon atoms, and is exemplified by $-CH_2CH_2-$, $-CH_2CH_2CH_2-$, $-CH(CH_3)CH_2-$, $-(CH_2)_4-$, and $-(CH_2)_5-$. The groups R^1 within the molecule may be identical or different. X is an alkoxy group having 1 to 4 carbon atoms, and it is exemplified by methoxy, ethoxy, propoxy, and methoxyethoxy.

The value of a is to be 2 or 3.

G is a group having the formula $-R^1O(C_2H_4O)_b(C_3H_5O)_cR^2$, and it functions to impart antistaticity and hydrophilicity to the solid. R^2 is to be the hydrogen atom or a monovalent organic group having 1 to 5 carbon atoms, and said monovalent hydrocarbon groups are exemplified by alkyl groups such as methyl, ethyl, and propyl, and by acyl groups such as acetyl and propionyl. In each G group b and c are both integers having values of 0 to 50 wherein the sum $b + c$ is to have a value of 2 to 100.

In the organopolysiloxane x is an integer having a value of 5 to 500, y is an integer having a value of 0 to 100, and z is an integer having a value of 0 to 100, with the condition that when A consists entirely of Q, z is then to be an integer having a value of 1 to 100. When x is 50, lubricity will also be imparted to the solid.

The organopolysiloxane to be used in the present invention can be synthesized, for example, by addition reacting diorganohydrogensilyl-terminated diorganosiloxane-organohydrogensiloxane copolymer with alkoxy-silyl group-containing alkene and alkenyl-substituted polyoxyalkylene compound, the latter two being used in the appropriate ratio, under the catalytic activity of a platinum-type catalyst such as chloroplatinic acid. Further synthesis details are disclosed in the examples below.

With regard to the use of the present invention's agent for treating solids, the above-described organopolysiloxane can be used as is, or it may be dissolved in water as is, or it may be auto-emulsified in water. Alternatively, it may be emulsified using a suitable emulsifying agent such as, for example, the salts of sulfate esters of higher alcohols, alkylbenzenesulfonate salts, higher alcohol-polyoxyalkylene adducts, alkylphenol-polyoxyalkylene adducts, higher fatty acid sorbitan esters, etc.

Alternatively, the solid treatment agent of the present invention may be used by dissolving the above-described organopolysiloxane in an organic solvent such as, for example, toluene, xylene, benzene, n-hexane, heptane, acetone, methyl ethyl ketone, methyl isobutyl ketone, ethyl acetate, butyl acetate, mineral terpene, perchloroethylene, trichloroethylene, etc.

Solids may be treated with the treatment agent of the present invention by methods such as spraying, roll coating, brush coating, immersion, etc. While the quantity of adhesion will vary with the type of solid and so cannot be strictly specified, it will generally be 0.01 to 10.0 wt % based on the solid. After application, a durable hydrophilicity and antistaticity will be imparted to the solid, for example, upon standing at room temperature, or upon blowing on hot air, or upon a heat treatment.

Furthermore, treatment may be conducted using the treatment agent of the present invention in combination with the metal salts of organic acids, for example, their zinc, tin, zirconium, etc., salts such as zinc stearate, zinc oleate, dibutyltin diacetate, dibutyltin dioleate, dibutyltin dilaurate; zirconium stearate; and/or amino-containing alkoxy-silanes, epoxy-containing alkoxy-silanes, organohydrogenpolysiloxanes, silanol-containing organopolysiloxanes; etc.

Solids which may be treated with the treatment agent of the present invention are exemplified by various fibers and their fabrics; sheet-form materials such as paper, natural and synthetic leathers, cellophane and plastic films; foams such as synthetic resin foams; synthetic resin moldings; natural and synthetic rubber moldings; metal moldings; glass moldings; and powders such as inorganic powders and synthetic resin powders.

The aforesaid fibers are exemplified in terms of species by natural fibers such as hair, wool, silk, flax, cotton and asbestos; by regenerated fibers such as rayon and acetate; by synthetic fibers such as polyester, polyamide, vinylon, polyacryl nitrile, polyethylene, polypropylene and spandex; and by glass fibers; carbon fibers; and silicon carbide fibers. They are exemplified in terms of form by the staple, filament, tow, and yarn. Their fabrics are exemplified by knits, nonwovens, resin-finished fabrics, and sewn articles thereof.

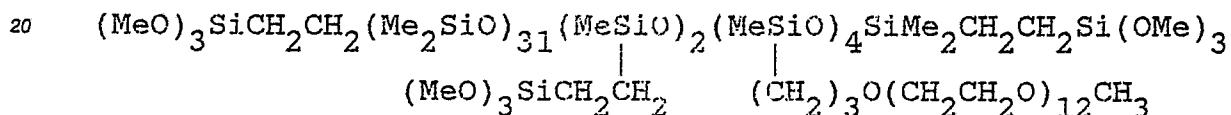
The invention will be explained in the following using illustrative examples. In the examples and reference examples, parts = weight parts and % = weight %, and the viscosity is the value measured at 25°C.

Reference Example 1

Vinyltrimethoxysilane, 148.2 g, is placed in a three-neck 500 ml flask equipped with a reflux condenser, the internal temperature is raised to 60°C by heating, 0.15 g 2% isopropanolic chloroplatinic acid solution is added, and 51.8 g tetramethyldisiloxane is dripped in. Organosiloxane I having the following formula is obtained by a reaction at 100°C for 2 hours and distillation in vacuo at 140°C/5 mmHg.

$(\text{CH}_3\text{O})_3\text{SiCH}_2\text{CH}_2\text{Si}(\text{CH}_3)_2\text{OSi}(\text{CH}_3)_2\text{CH}_2\text{CH}_2\text{OCH}_3$ Organosiloxane I, 61.4 g, methylhydrogensiloxane cyclic tetramer, 59.8 g, cyclic dimethylsiloxane tetramer, 368.9 g, and 15 g thoroughly dried activated clay as polymerization catalyst are placed in a three-neck 500 ml flask equipped with a reflux condenser, and polymerized at 75°C for 10 hours. After cooling, filtration is conducted using an assistant.

Exactly 136.9 g of this organopolysiloxane product, 13.4 g vinyltrimethoxysilane, 149.7 g allyl-containing polyether having the following formula $\text{CH}_2=\text{CHCH}_2\text{O}(\text{CH}_2\text{CH}_2\text{O})_{12}\text{CH}_3$ and 90 g toluene are placed in a 500 ml three-neck flask equipped with a reflux condenser, the internal temperature is raised to 60°C by heating, 0.38 g 2% isopropanolic chloroplatinic acid solution is added, and a reaction is then carried out at 120°C for 2 hours. After the completion of the reaction, the volatiles are stripped in vacuo. The product is identified by infrared absorption spectral analysis and nuclear magnetic resonance analysis as an organopolysiloxane (Organopolysiloxane A, viscosity = 305 cS) having the following formula:

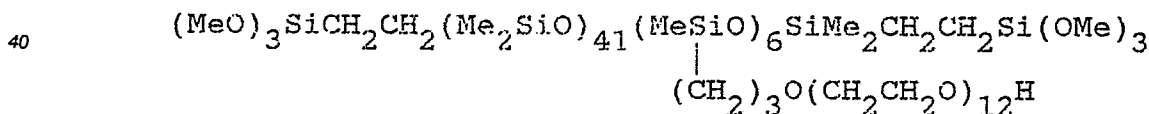


Reference Example 2

Siloxane II, 89.1 g, having the formula



7.5 g vinyltrimethoxysilane, and 60 g toluene are placed in a 500 ml three-neck flask equipped with a reflux condenser, the internal temperature is raised to 80°C by heating, 0.1 g 2% isopropanolic chloroplatinic acid solution is added, and a reaction is conducted at 110°C for 30 minutes. After cooling to 80°C, 103.4 g allyl-containing polyether I having the formula $\text{CH}_2=\text{CHCH}_2\text{O}(\text{CH}_2\text{CH}_2\text{O})_{12}\text{H}$ is added, the temperature is raised to 90°C, 0.15 g 2% isopropanolic chloroplatinic acid solution is added, and a reaction is conducted at 120°C for 1 hour. After the end of the reaction, the volatiles are stripped in vacuo at 140°C/5 mmHg. Infrared absorption spectral analysis and nuclear magnetic resonance analysis confirm the product to be an organopolysiloxane (Organopolysiloxane B, viscosity = 1,100 cS) having the following formula:

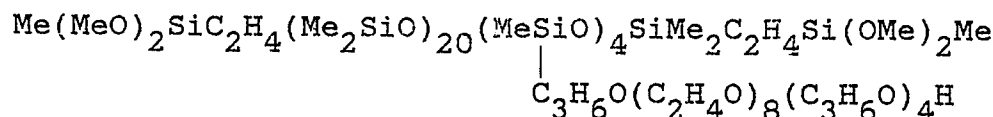


Reference Example 3

A siloxane, 69.3 g, having the formula



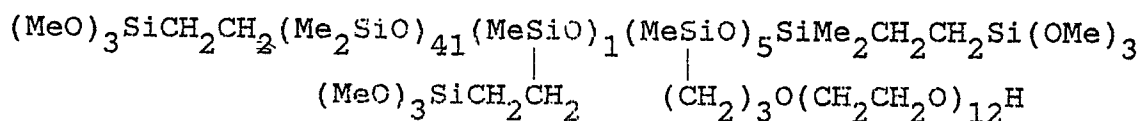
11.0 g methylvinyltrimethoxysilane and 60 g toluene are placed in a 500 ml three-neck flask equipped with a reflux condenser, the internal temperature is raised to 80°C by heating, 0.1 g 2% isopropanolic chloroplatinic acid solution is added, and a reaction is carried out for 30 minutes at 105°C. After cooling to 80°C, 119.7 g allyl-containing polyether having the formula $\text{CH}_2=\text{CHCH}_2\text{O}(\text{C}_2\text{H}_4\text{O})_8(\text{C}_3\text{H}_6\text{O})_4\text{H}$ is added, the temperature is raised to 90°C, 0.15 g 2% isopropanolic chloroplatinic acid solution is added, and a reaction is conducted at 120°C for 1 hour. After the end of the reaction, the volatiles are stripped in vacuo at 140°C/5 mmHg. Infrared absorption spectral analysis and nuclear magnetic resonance analysis confirm the product to be an organopolysiloxane (Organopolysiloxane C, viscosity = 500 cS) having the following formula:



Reference Example 4

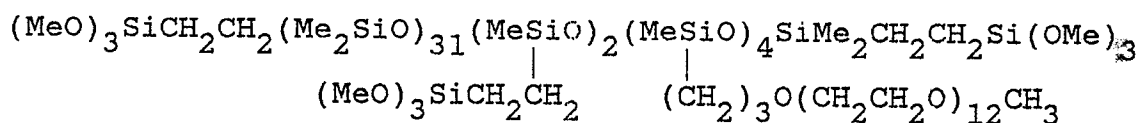
Siloxane II, as used in Reference Example 2, 89.1 g vinyltrimethoxysilane, 7.5 g, allyl group-containing polyether I as used in Reference Example 2, 103.4 g, and 60 g toluene are placed in a three-neck 500 ml flask equipped with a reflux condenser, the internal temperature is raised to 80°C by heating, 0.25 g 2% isopropanolic chloroplatinic acid solution is added, and a reaction is conducted at 125°C for 1 hour. After the reaction, the volatiles are stripped in vacuo at 140°C/5 mmHg.

Infrared absorption analysis and nuclear magnetic resonance analysis confirm this material to be an organopolysiloxane (Organopolysiloxane D, viscosity = 500 cS) having the following structure:



Example 1

One part of the Organopolysiloxane A synthesized according to Reference Example 1 and having the formula



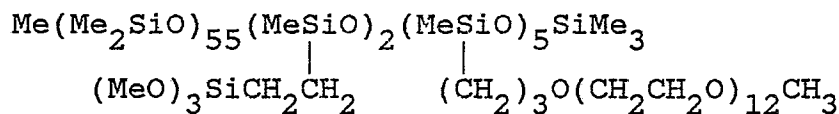
8 parts glyoxal-type resin (Sumitex Resin NS-2 from Sumitomo Chemical Co., Ltd.), 2 parts amine catalyst (Sumitex Accelerator X-80 from Sumitomo Chemical Co., Ltd.), 0.5 part of a tin catalyst (50% emulsion of dibutyltin dilaurate), and 88.9 parts water are mixed to homogeneity to prepare a treatment bath. Two sheets of twilled fabric (40 cm x 20 cm, 65% polyester/35% cotton) are immersed in this treatment bath for 30 seconds, adjusted to a 100% expression ratio on a mangle, dried at room temperature for 10 hours, and then heated in an oven at 150°C for 5 minutes. The resulting organopolysiloxane-finished fabrics are then each cut in half. One sheet of the resulting four fabric sheets is washed once, one sheet is washed 5 times, and one sheet is washed 10 times (wash conditions as below). Following this, these are rinsed twice with water (under the wash conditions, but with no detergent) to afford washed fabrics. Bath ratio = 1/50: Time = 10 minutes: Temperature = 40°C. Detergent = 0.5% aqueous solution of New White (from Lion Corp.)

In the water absorptivity test, the organopolysiloxane-finished fabric (before and after washing) is laid out horizontally on spread out filter paper, a drop of water is delivered from a syringe, and the time required for its soaking in is then measured.

To measure the residual organopolysiloxane, the % residual organopolysiloxane after washing is analyzed using an X-ray fluorescent analyzer from Rigaku Corp. based on the difference in silicon atom counts before and after washing the treated fabric. The results are reported in Table 1.

Comparison Example 1

Treatment is conducted exactly as in Example 1, with the exception that 1 part Organopolysiloxane (i) having a viscosity of 1,200 cS and the formula



is used in place of Organopolysiloxane A. Testing is conducted as in Example 1, and these results are also reported in Table 1.

Table 1

Water Absorbitivity				% Residual			
(seconds)				Organopolysiloxane			
Number of Washes				Number of Washes			
0	1	5	10	0	1	5	10

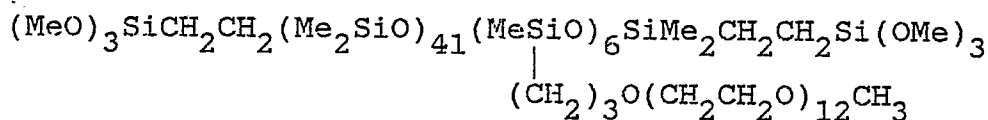
Example 1	6.3	4.4	2.3	2.0	100.0	94.5	87.0	85.0
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Comparison

Example 1	17.5	12.2	9.3	6.6	100.0	81.8	72.2	66.0
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Example 2

Five parts of Organopolysiloxane B, synthesized according to Reference Example 2 and having the formula



N-(beta-aminoethyl)-gamma-aminopropyltrimethoxysilane, 0.5 part, dibutyltin diacetate, 0.2 part and 99.5 parts toluene are mixed to homogeneity to prepare a treatment bath. 65% polyester/35% cotton broadcloth (40 cm x 20 cm), which has been coated with a 3% add-on of the glyoxal-type resin as used in the treatment bath in Example 1, is immersed in this treatment bath for 30 seconds, adjusted to an expression ratio of 100% using a mangle, dried at room temperature by standing for 10 hours, and then heated in an oven at 150°C for 5 minutes. The obtained organopolysiloxane-finished fabric is cut in half, and one sample is washed once under the wash conditions of Example 1 and rinsed twice with water to afford a washed sample.

Both before and after washing, the organopolysiloxane-finished fabric is subjected to the water absorbitivity test and testing of the % residual organopolysiloxane as described in Example 1. These results are reported in Table 2.

Comparison Example 2

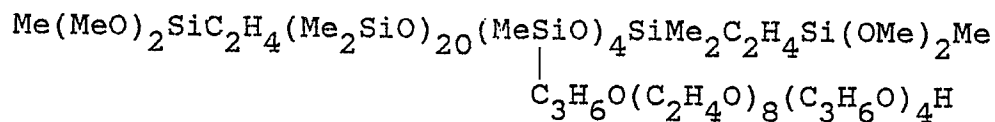
Treatment and testing are conducted by the methods described in Example 2, with the exception that Organopolysiloxane (i), as used in Comparison Example 1, is used instead of Organopolysiloxane B. These results are also reported in Table 2.

Table 2

		Water Absorbitivity	% Residual
		(seconds)	Organopolysiloxane
		Before Wash	After Wash
5			
10	Example 2	4.5	60
	Example 3	4.3	45
15	Comparison		
	Example 2	5.0	55
20	Comparison		
	Example 3	3.1	11

25 Example 3

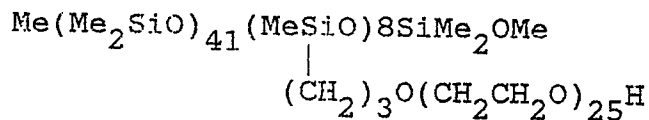
Treatment and testing are conducted by the methods of Example 2, with the exception that 5 parts Organopolysiloxane C (viscosity = 500 cS), synthesized as in Reference Example 3 and having the following formula



is used in place of organopolysiloxane B. These results are reported in Table 2.

Comparison Example 3

Treatment and testing are conducted as described in Example 2, with the exception that 5 parts Organopolysiloxane (ii) having a viscosity of 1,000 cS and the formula



is used in place of Organopolysiloxane B. These results are reported in Table 2.

Example 4

Organopolysiloxane A as used in Example 1, 5 parts, is dissolved to homogeneity in 995 parts toluene. A 65% polyester/35% cotton broadcloth (40 x 20 cm), coated with a 3% add-on of the glyoxal-type resin, is then immersed in this solution for 30 seconds, adjusted to an expression ratio of 100% on a mangle, dried by standing at room temperature for 10 hours, and then heated in an oven for 5 minutes at 150°C. The obtained organopolysiloxane-finished fabric is then cut in half, and one sample of organopolysiloxane-finished fabric is washed once under the wash conditions of Example 1 and rinsed twice. Test samples are thus prepared of organopolysiloxane-finished fabric before and after washing.

An antistaticity test is carried out as follows. The treated fabrics (before and after washing) are allowed to stand at 20°C/RH 65% for 1 week. Their triboelectrification voltages are then measured using a Kyodai Kaken rotary static tester (800 rpm for 60 seconds). Cotton fabric (unbleached muslin #3) is used as the friction fabric.

A soiling resistance test is conducted as follows. An artificial soilant is prepared by thoroughly mixing and pulverizing 300 g ASTM No. 1 oil, 3 g coal tar, 5 g dried clay powder, 5 g portland cement and 5 g sodium dodecylbenzenesulfonate in a mortar. 5 ml of this artificial soilant, 100 ml of a 0.5% aqueous solution of Marseilles soap and 10 steel balls are placed in a 450 ml glass bottle. Washed and unwashed samples of organopolysiloxane-finished fabric and untreated fabric are respectively placed in the bottle, treated at 60°C for 30 minutes, and then gently rinsed with water and dried. These are then washed for 10 minutes on high in an automatic reversing rotary washing machine using a 0.5% aqueous solution of Marseilles soap. After then washing in water and drying, the reflectance of the test fabrics is measured at a wavelength of 550 millimicrons using a reflectometer. These test results are reported in Table 3.

Comparison Example 4

Treatment and testing are conducted by the methods of Example 4, but using 5 parts of the Organopolysiloxane (ii) used in Comparison Example 3 in place of Organopolysiloxane A. These results are also reported in Table 3.

Table 3

	Triboelectrification		% Reflectance at
	<u>Voltage (V)</u>		<u>550 millimicrons</u>
	Before	After	
	<u>Wash</u>	<u>Wash</u>	
Example 4	880	1030	71
Comparison			
Example 4	900	1530	53
Untreated			
Fabric	1650	1610	53

Example 5

Organopolysiloxane A as synthesized in Reference Example 1, 10 parts, and 1 part zinc stearate are dissolved to homogeneity in 89 parts water to prepare a treatment solution. This is then applied at an organopolysiloxane add-on of 0.2 g/m² by spraying on one side of a plasma-processed polyethylene terephthalate film. After coating, the film is dried at room temperature overnight and then heated in an oven at 130°C for 10 minutes.

In the comparison examples, 10% aqueous solutions are prepared using, respectively, Organopolysiloxane (ii) (used in Comparison Example 3) or nonionic surfactant (NS-210 from Nippon Oils and Fats Co., Ltd.), and each is then sprayed on one side of the plasma-processed polyethylene terephthalate film at an add-on of 0.2 g/m², followed by drying and heating.

The obtained three treated films are immersed in running water for 6 hours. After this, the upper surface of a thermostatted water bath (set at 60°C ± 2°C) is covered and sealed with each film, wherein the treated surface is down. The status of the films is inspected after 3 hours. The film treated with Organopolysiloxane A, a treatment agent of the present invention, is hydrophilic, presents uniform wetting of the lower film surface, and is transparent. However, the lower surfaces of the other 2 films are not hydrophilic, and are adhered with water droplets and are opaque.

Example 6

A 0.5% aqueous solution of Organopolysiloxane D, 100 g, (synthesized in Reference Example 4) is prepared, 50 g carbon black powder is added, and this mixture is allowed to dry by standing. Heating at 100°C for 5 minutes then affords a carbon black powder having a 1% add-on of Organopolysiloxane D.

In the comparison example, carbon black powder is similarly treated with Organopolysiloxane (ii) to afford a carbon black powder having a 1% add-on of Organopolysiloxane (ii).

50 g of each carbon black powder is separately placed in 1 l of water, followed by stirring for 3 hours, filtration and drying. 5 Parts of each of the obtained carbon black powders is then separately dispersed to homogeneity in an aqueous acrylic emulsion paint to produce a paint. The paint containing the Organopolysiloxane D-treated carbon black powder presented a uniform dispersion, and did not suffer from sedimentation. However, the Organopolysiloxane (ii)-treated carbon black powder underwent rapid sedimentation, and the dispersion was inhomogeneous. These observations indicate that the present invention's agent for treating solids has the capacity to impart a durable hydrophilicity.

Because the present invention's agent for treating solids has as its principal agent an organopolysiloxane which has the alkoxysilylalkyl group at least at one molecular terminal and which also has at least 1 polyoxyalkylene group as a pendant group or at the molecular terminals, it can impart a durable hydrophilicity and antistaticity to solid materials. As a consequence, it is very useful industrially.

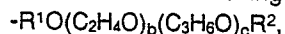
Claims

1. A composition for treating solids, said composition comprising an organopolysiloxane compound which has the formula $A(R_2SiO)_x(RQSiO)_y(RGSiO)_zSiR_2A$, wherein

Q denotes a radical having the formula



G denotes a radical having the formula



A denotes a radical selected from the group consisting of Q and G radicals, at least one A radical being a Q radical,

x has a value of from 5 to 500,

y has a value of from 0 to 100,

z has a value of from 0 to 100,

X denotes an alkoxy or alkoxyalkoxy radical having from 1 to 4 carbon atoms,

R denotes a monovalent hydrocarbon or halogenated hydrocarbon radical having from 1 to 10 carbon atoms,

R¹ denotes an alkylene radical having from 2 to 5 carbon atoms,

R² denotes a hydrogen atom or a monovalent organic radical having from 1 to 5 carbon atoms,

a has a value of 2 or 3,

b has a value of from 0 to 50,
c has a value of from 0 to 50, and
b plus c has a value of from 2 to 100;

there being present in said organopolysiloxane at least one G radical.

- 5 2. A composition according to claim 1 further comprising a curing amount of a curing agent comprising a curing catalyst and/or a crosslinking compound for silanol groups.
3. A composition according to claim 1 further comprising a glyoxal resin.
4. A method for treating a solid, said method comprising applying the composition of claim 1 to the solid and heating the composition-containing solid to a temperature of from 50 to 150°C.
- 10 5. A method for improving the hydrophilicity of a fabric containing polyester fibers said method comprising applying the composition of claim 1 to the fabric and heating the composition-containing fabric to a temperature of from 50 to 150°C.
6. A treated solid provided by the method of claim 4.
7. A treated fabric provided by the method of claim 5

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