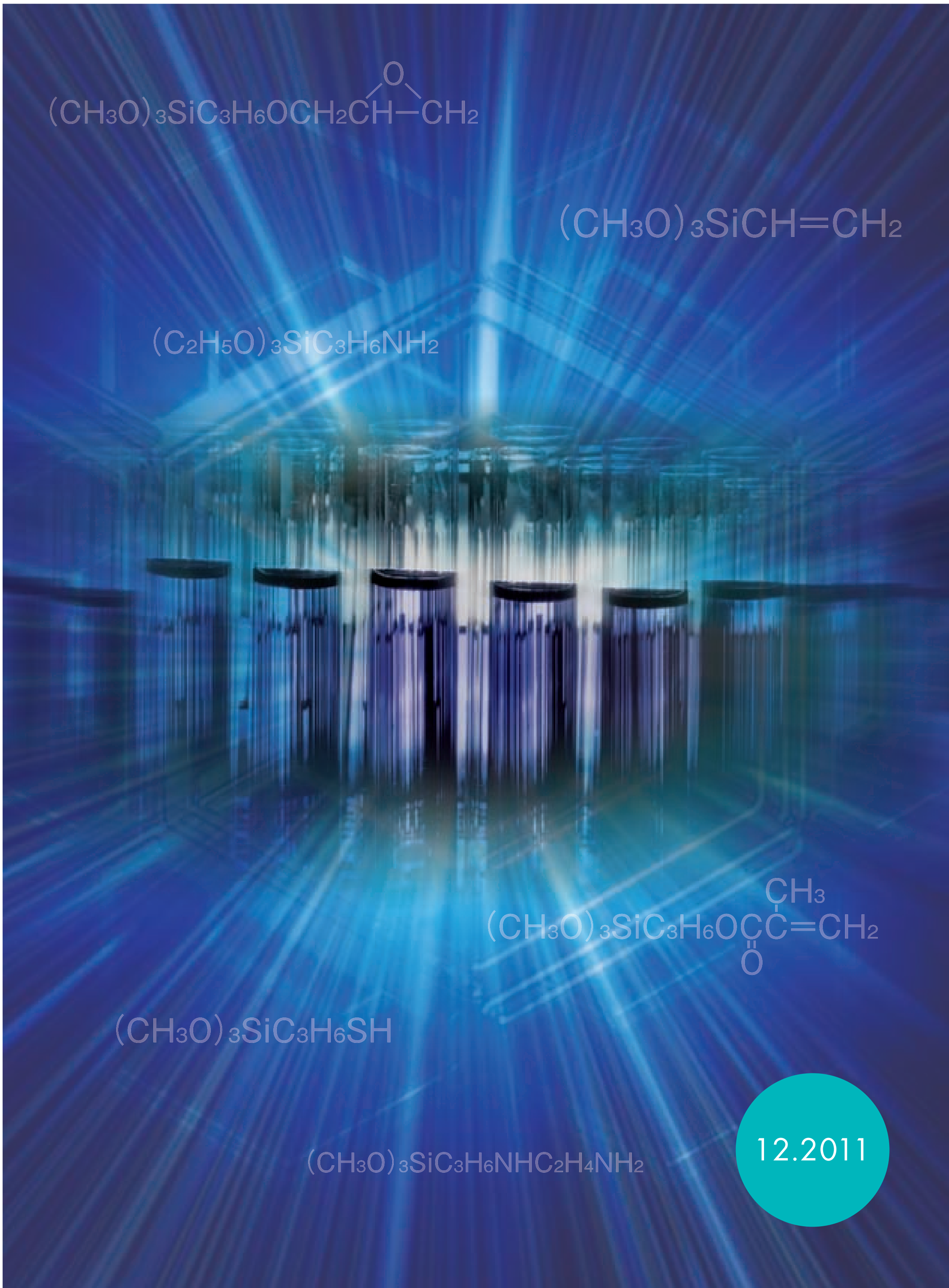
Shin-Etsu Silicone

Silane Coupling Agents



Silanes
Silane Coupling Agents
Alkoxy Oligomers

Our diverse array of materials enable users to enhance the quality and functionality of their products, and expand the possibilities for new product development.

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Product Outlines

Silane Coupling Agents

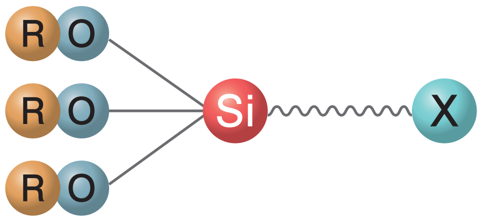
Silane coupling agents are compounds whose molecules contain functional groups that bond with both organic and inorganic materials.
A silane coupling agent acts as a sort of intermediary which bonds organic materials to inorganic materials.
It is this characteristic that makes silane coupling agents useful for improving the mechanical strength of composite materials, for improving adhesion, and for resin modification and surface modification.

Molecular Structure

The individual molecules of silane coupling agents contain two types of reactive functional groups characterized by different kinds of reactivity.

RO: Reactive groups that form chemical bonds with inorganic materials including glass, metals, silica stone

• Methoxy groups • Ethoxy groups, other



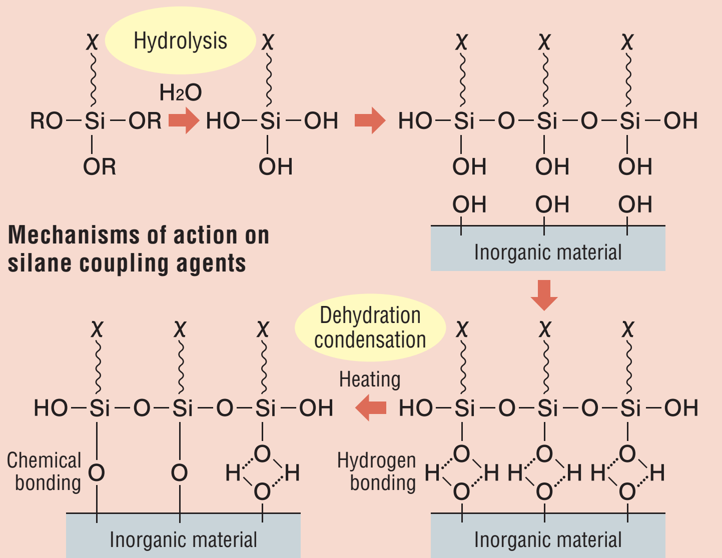
X: Reactive groups that form chemical bonds with organic materials such as synthetic resins

• Vinyl groups • Epoxy groups • Amino groups
• Methacryloxy groups • Mercapto groups, other

Mechanism of action on inorganic and metallic materials

Silane coupling agents react with water (hydrolysis) to form silanol groups, and oligomers are formed through partial condensation.
The silanol oligomers then hydrogen bond to the surface of the inorganic material.
Finally, the inorganic material put through a drying process and robust chemical bonds are formed through a dehydration condensation reaction.

■ **Mechanisms of action on silane coupling agents**



Mechanism of action on organic materials

When a silane coupling agent is used for surface treating an inorganic material to promote adhesion between the inorganic material and an organic material, it acts in the following ways.

- **By improving the wettability of resins**
- **By improving miscibility in resins**
- **By forming chemical bonds with resins**
- **By hydrogen bonding with resins**

With thermoplastic resins

Silane coupling agents are effective with thermoplastic resins of relatively high polarity.

With heat-curable resins

Silane coupling agents are effective for improving the strength of heat-curable resins, provided that the silane coupling agent contains organic functional groups which can be involved in the curing reaction of the resin.

Alkoxy Oligomers

The molecules of alkoxy oligomers include organic functional groups as well as alkoxy groups (similar to silane coupling agents).
Alkoxy oligomers are more multifunctional than silanes, and can be used as an oligomeric coupling agent for resin modification or as a functional coating agent.

Silanes

These are alkoxy silanes whose molecules contain methyl groups, long-chain alkyl groups, phenyl groups and other organic groups.
Silanes are used in a wide variety of fields; they can be used for surface modification of inorganic materials to impart water repellency and have many other uses.
In addition to our alkoxy silanes, Shin-Etsu also offers a line of silazanes.

Silylating Agents

Silylating agents are used in organic synthesis in the manufacture of pharmaceuticals and agrichemicals, and in electronics manufacturing.
Silylating agents can be used to introduce organosilyl groups into organic or inorganic materials where they act as protecting groups for active hydrogens.
Different types of alkyl groups vary in their bulkiness and ease of separation; by using the proper alkyl groups, the user can gain better control over the organic synthesis reaction.

Silane Coupling Agents

Each molecule of a silane coupling agent contains two types of functional groups, namely organic functional groups and alkoxy groups.

■ General properties

Functional group	Product name	Chemical name	Molecular weight	Specific gravity 25°C
Vinyl	**KBM-1003**	Vinyltrimethoxysilane	148.2	0.97
	KBE-1003	Vinyltriethoxysilane	190.3	0.90
Epoxy	**KBM-303**	2-(3,4 epoxycyclohexyl) ethyltrimethoxysilane	246.4	1.06
	KBM-403	3-Glycidoxypropyl trimethoxysilane	236.3	1.07
	KBE-402	3-Glycidoxypropyl methyldiethoxysilane	248.4	0.98
	KBE-403	3-Glycidoxypropyl triethoxysilane	278.4	1.00
Styryl	**KBM-1403**	p-Styryltrimethoxysilane	224.3	1.06
Methacryloxy	**KBM-502**	3-Methacryloxypropyl methyldimethoxysilane	232.4	1.00
	KBM-503	3-Methacryloxypropyl trimethoxysilane	248.4	1.04
	KBE-502	3-Methacryloxypropyl methyldiethoxysilane	260.4	0.96
	KBE-503	3-Methacryloxypropyl triethoxysilane	290.4	0.99
Acryloxy	**KBM-5103**	3-Acryloxypropyl trimethoxysilane	234.3	1.06
Amino	**KBM-602**	N-2-(Aminoethyl)-3-aminopropylmethyldimethoxysilane	206.4	0.97
	KBM-603	N-2-(Aminoethyl)-3-aminopropyltrimethoxysilane	222.4	1.02
	KBM-903	3-Aminopropyltrimethoxysilane	179.3	1.01
	KBE-903	3-Aminopropyltriethoxysilane	221.4	0.94
	KBE-9103	3-Triethoxysilyl-N-(1,3 dimethyl-butylidene) propylamine	—	0.92
	KBM-573	N-Phenyl-3-aminopropyltrimethoxysilane	255.4	1.07
	KBM-575	N-(Vinylbenzyl)-2-aminoethyl-3-aminopropyltrimethoxysilane hydrochloride	—	0.91
	KBM-974	N-(Vinylbenzyl)-2-aminoethyl-3-aminopropyltrimethoxysilane hydrochloride, hydrolysate	—	0.91
Ureide	**KBE-585**	3-Ureidopropyltriethoxysilane	—	0.91
Mercapto	**KBM-802**	3-Mercaptopropylmethyldimethoxysilane	180.3	1.00
	KBM-803	3-Mercaptopropyltrimethoxysilane	196.4	1.06
Sulfide	**KBE-846**	Bis(Triethoxysilylpropyl)tetrasulfide	—	1.08
Isocyanate	**KBE-9007**	3-Isocyanatepropyltriethoxysilane	247.4	1.00

✻ 1kPa: 7.5 mmHg

■ Primers These primers contain one or two types of silane coupling agents diluted with a solvent.

■ Typical primers

Product name	Appearance	Features	Active content %
KBP-40	Colorless to pale yellow transparent liquid	Aminosilane. Excellent water resistance	7
KBP-43	Colorless to pale yellow transparent liquid	Aminosilane. Excellent water resistance and weatherability	21
KBP-44	Colorless to pale yellow transparent liquid	Isocyanate silane	14
X-12-414	Colorless to pale yellow transparent liquid	Mercapto silane	15
KBP-90	Colorless to yellow liquid	Amino silane-water solution	32

	Refractive index 25°C	Boiling point °C	Flash point °C	Minimum covering area m^2/g	UN hazard classification	Existing substances No.	CAS No.
	1.391	123	23	515	UN-1993	2-2066	2768-02-7
	1.397	161	54	410	UN-1993	2-2066	78-08-0
	1.448	310	163	317	Not applicable	3-2647	3388-04-3
	1.427	290	149	330	UN-3082	2-2071	2530-83-8
	1.431	259	128	314	Not applicable	2-2072	2897-60-1
	1.425	124°C/0.39 kPa	144	280	Not applicable	2-2071	2602-34-8
	1.504	115°C/0.001 kPa	136	348	Not applicable	3-4371	18001-13-3
	1.433	83°C/0.39 kPa	115	335	Not applicable	2-2075	14513-34-9
	1.429	255	125	314	Not applicable	2-2076	2530-85-0
	1.432	265	136	300	Not applicable	2-2075	65100-04-1
	1.427	129°C/0.67 kPa	128	270	Not applicable	2-2076	21142-29-0
	1.427	102°C/0.53 kPa	115	333	Not applicable	2-3727	4369-14-6
	1.447	234	110	380	Not applicable	2-2084	3069-29-2
	1.442	259	128	351	UN-3082	2-2083	1760-24-3
	1.422	215	88	436	Not applicable	2-2061	13822-56-5
	1.420	217	98	353	Not applicable	2-2061	919-30-2
	1.437	—	134	—	Not applicable	7-2673	—
	1.504	312	165	307	Not applicable	3-2644	3068-76-6
	—	—	11	—	UN-1992	3-3378	34937-00-3
	—	—	11	—	UN-1992	—	171869-90-2
	—	—	11	—	UN-1992	2-2968	23779-32-0
	1.448	204	72	432	UN-3082	2-3498	31001-77-1
	1.440	219	107	398	UN-3082	2-2045	4420-74-0
	1.486	—	212	—	Not applicable	2-3124	40372-72-3
	1.418	250	118	315	UN-2927	2-3880	24801-88-5

(Not specified values)

	Typical diluents	UN hazard classification
	Ethanol	UN-1133
	Toluene, xylene, ethyl acetate	UN-1133
	Toluene, ethyl acetate	UN-1133
	Toluene, ethyl acetate	UN-1992
	Water	—

(Not specified values)

Preparation of a silane hydrolysate solution

Silane coupling agents are typically used in the form of a dilute aqueous solution. The silane coupling agent contains hydrolyzable alkoxy groups which react with inorganic materials. When these groups are hydrolyzed, silanol groups are formed which adhere and bond to hydroxyl groups on the surface of the inorganic material. The hydrolyzable alkoxy groups include methoxy and ethoxy groups. Methoxy groups and ethoxy groups have different hydrolytic properties, and are thus used for different types of applications.

Preparing the solution

Silane coupling agent concentration

Concentration should generally be around 0.1–2.0%. Dilute with water or a water-alcohol solution.

Adjusting pH

If the silane coupling agent does not dissolve readily in water, the pH can be adjusted to slight acidity with acetic acid. A 0.1–2.0% acetic acid-water solution will have the effect of promoting the hydrolysis of the silane coupling agent and stabilizing the formed silanol in the solution. It may not be necessary to adjust the pH of amino silanes; see page 18 (Solubility in water) for more information.

Drip-feed method

While stirring the aqueous solution, gradually add drops of the silane coupling agent. Stir briskly, but not so fast that the liquid splashes out. If the silane coupling agent solution is added all at once, there may be problems of poor dispersion and formation of colloidal particles. After the silane has been dripped in, continue stirring for 30–60 minutes. When the solution becomes transparent, it indicates that hydrolysis is nearly complete.

Filtering the aqueous solution

If you notice undissolved or floating matter in the solution, it may be best to filter it. When using a silane-water solution in a continuous process, filtration is recommended using a circulating filter system that has a cartridge with a pore size of 0.5 μm or smaller.

✻ Silane coupling agents will dissolve in almost any organic solvent, but be aware that using ethanol with methoxy types or methanol with ethoxy types may result in an exchange reaction.

Solution preparation

Treating inorganic materials

The most effective method of treating with silane coupling agents involves treating the inorganic material first, prior to mixing it with the organic material.
Generally speaking, the way silane coupling agents work is that the alkoxy groups are hydrolyzed to form silanol groups that bond chemically to the inorganic material.
But on certain metals, the organic functional groups (e.g. mercapto and sulfide groups) may also be involved in the reaction.

■ Effectiveness of silane coupling agents for various inorganic materials

Degree of effectiveness	Inorganic material
Highly effective	Glass, silica, aluminum oxide
Effective	Talc, clay, aluminum, aluminum hydroxide, mica
Slightly effective	Asbestos, titanium oxide, zinc oxide, iron oxide
Almost no effect	Graphite, carbon black, calcium carbide

Treatment of inorganic materials generally involves one of two things.
1) Surface treating inorganic fibers and metal foils
2) Treating fillers

Inorganic fibers and metal foils

Densely packed fibrous materials such as those in inorganic fabrics should be heat cleaned or washed with warm water before using the silane coupling agent.
With metal foils, first check the wetting properties of the material and choose a solvent for dilution if needed.

Wash the substrate
↓
Dip in the hydrolysate solution
↓
Pull out substrate and remove excess liquid
↓
Dry (100–110 °C)

- The treatment is highly effective.
- Even treatment is possible; this type of treatment is used extensively in industrial applications.

Inorganic fillers and other materials

The inorganic material is surface treated with a silane coupling agent before being added to an organic resin. This can be done using a dry treatment or wet treatment method.
The wet method enables even treatment, but the waste fluid must be processed. For this reason, the dry method is better in terms of productivity.

Dry method

In this method, the silane is added at full strength and evenly dispersed in the filler as it is stirred at high speed with a mixer. Although it can be difficult to achieve even treatment with this method, it is widely used in industrial applications because large amounts of filler can be treated in a short time. When treating dry fillers, the treatment process will be more efficient if water is added first. Add around 1% by weight of water to the filler and let it sit for around 8 hours, keeping the container closed (minimize contact with air) and at a temperature of 50–70 °C. This promotes even dispersion of the water.

Stir filler with mixer, etc.
↓
Add silane coupling agent by drip or spray
↓
Dry

- Productivity is high.
- Clumping may occur in some cases.

Wet method

In this method, the filler is immersed in a dilute silane solution. Alkyl silanes (especially long-chain alkyl silanes and fluoroalkyl silanes) are highly hydrophobic, and so treatment is difficult in systems using water alone (including acetic acid-water). Treatment with alkoxy silanes should thus be done using a water-alcohol solution whose pH has been properly adjusted using acetic acid or other agent.
The surfaces of the filler particles can be evenly treated, which allows for a highly precise treatment.

Dip in a hydrolysate solution or add silane coupling agent to a slurry
↓
Filler → Waste fluid
↓
Dry

- Enables even treatment.
- Productivity is low, because large amount of heat is required to remove water.
- Silane-containing waste fluid must be treated.

Treating organic resins

Addition to organic materials

This method is somewhat less effective than methods involving pre-treatment of the inorganic materials, yet it is widely used in industrial applications due to benefits gained in ease of handling and cost effectiveness.

Integral blending method

In this method, the silane coupling agent is added to the organic materials before the inorganic and organic materials are mixed. Adding amino silanes (KBM-602, KBM-603, KBM-903, KBE-903, KBM-573) to epoxy resins or phenolic resins may cause thickening or gelation, so keep in mind the effect this could have on pot life.

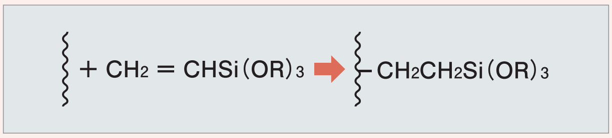
Master batch method

In this method, the silane coupling agent is first mixed with a small amount of the organic material to create a master batch which is then added to the other materials.

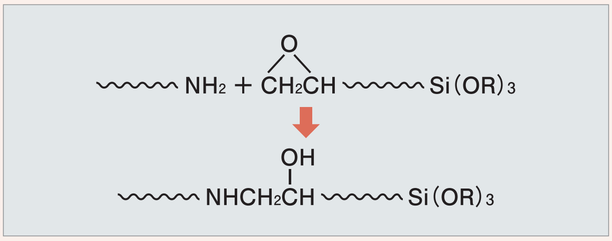
Reaction with organic resins (copolymerization)

For inorganic-organic hybrid materials (and others), a silane coupling agent can be reacted with a resin to make it a moisture-cure resin or impart other new types of functionality to the resin. Alkoxysilyl groups can be introduced through grafting or copolymerization.

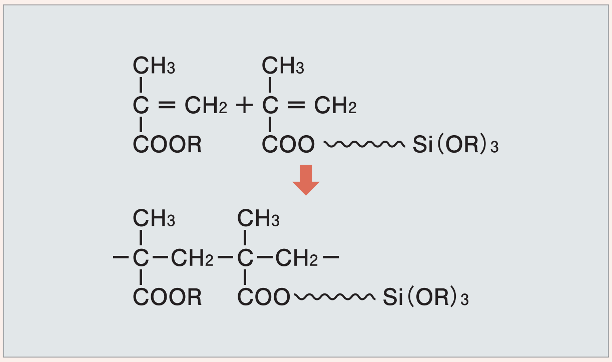
Grafting



Chemical reaction



Copolymerization



Organic functional groups and compatible resins

Functional groups \ Resin	Thermoplastic resins										Thermosetting resins								Elastomer/Rubber										
	Polyethylene	Polypropylene	Polystyrene	Acrylic	PVC	Polycarbonate	Nylon	Urethane	PBT & PET	ABS	Melamine	Phenolic	Epoxy	Urethane	Polyimide	Diallyl phthalate	Unsaturated polyester	Furan	Polybutadiene rubber	Polyisoprene rubber	Sulfur-crosslinked EPM	Peroxide Crosslinked EPDM	SBR	Nitrile rubber	Epichlorohydrin rubber	Neoprene rubber	Butyl rubber	Polysulfide	Urethane rubber
Vinyl	◎	◎														○	○				○	○							
Epoxy	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○					○	○	○		○	○	○
Styryl			○	○																									
Methacryloxy	◎	◎	◎	○		○		○		◎						○	◎				○	◎							
Acryloxy	○	○	○	○		○		○		◎						○	◎				○	◎							
Amino	○	○	◎	◎	◎	○	◎	○	○	○	○	◎	◎	○	○			◎			○	○		○		○	○	○	○
Ureide							◎					○		○	○														
Mercapto	○	○	○		○			○		○		○	○	○					○	○	◎	○	○	○	○	○		◎	◎
Sulfide																					◎		○	○	○	○		○	○
Isocyanate						○	○	◎	○	○	○	○	○	◎	○			○											○

◎: Very effective ○: Effective ✻ Not all the functional groups are capable of coupling with the resins in question. This should taken as a guide.

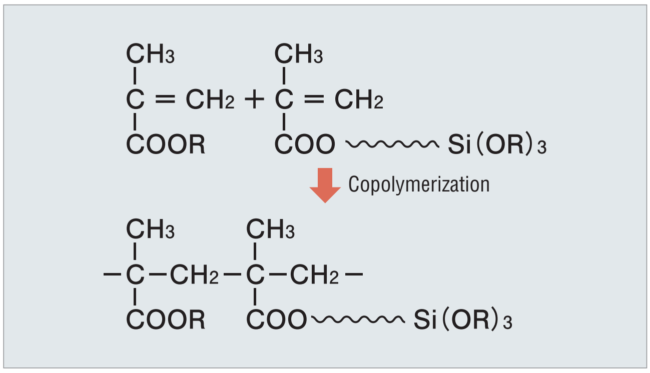
Examples of reactions involving organic functional groups

Amino group reactions (KBE-903)

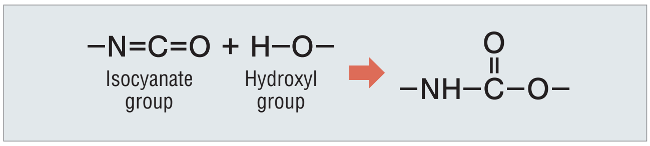
■ Reactions between amino groups and various functional groups

Amino group	Functional group	Reaction	
KBE-903 NH_2	Cl—	Dehydrochlorination reaction	NH—
	ClCO—	Amidation reaction	NHCO—
	O	Epoxide ring-opening reaction	NH, OH
	OCN—	Ureidation reaction	NHCONH—
	OH—	Hydrogen bonding with hydroxyl groups	H N H O
	H_2N—	Hydrogen bonding with amino groups	H N H N H
	$HOSO_2$—	Salt formed with sulfonic acid	$N^+H_3{}^-OSO_2$—
	HOOC—	Salt formed with carboxylic acid	$N^+H_3{}^-OOC$—

Methacryloxy group reactions (KBM-503)



Reaction between isocyanate groups & hydroxyl groups (KBE-9007)



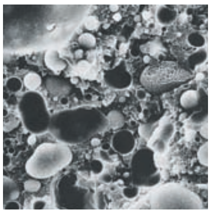
Application examples

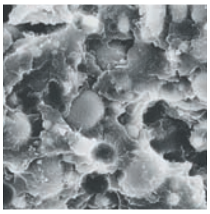
Silane coupling agents are vital to the development of higher quality materials with enhanced functionality.
They are put to use in an incredible array of products that help make modern living more comfortable and convenient.

Chemicals

- Silane coupling agents are used extensively in adhesives and paints to improve adhesion to inorganic substrates.
- Silane coupling agents are used to improve transparency and reduce viscosity during mixing and kneading.

Electron micrograph: Broken surface of unsaturated polyester resin compounded with spherical silica

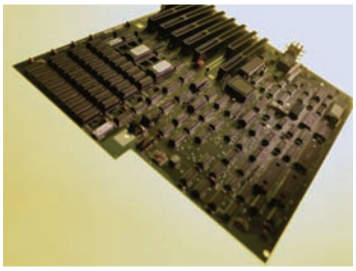

Not treated with silane coupling agent


Treated with silane coupling agent


Adhesives

Electrical applications

- Silane coupling agents play an essential role in improving the reliability of semiconductor encapsulants, flat-panel displays and other products.
- Silane coupling agents can improve the dispersibility and adhesion of inorganic materials.
- Silane coupling agents can be used as a raw material for hard-coat materials.


Electronic parts & circuit boards


LCD TVs & displays

Automotive applications

- Silane coupling agents are used to make silica reinforced tires and in development of new types of materials.
- Silane coupling agents can be used to create high-molecular-weight rubbers and silicas.
- Silane coupling agents are used to make highly weatherable paints.


Tires


Auto interiors & exteriors

Energy applications

- Silane coupling agents are essential to the manufacture of encapsulating resins for solar panels and hybrid materials which are used in wind power systems.


Solar cell modules


Wind power

Construction applications

- Silane coupling agents help improve the durability of exterior walls of homes and of bridges and other infrastructure.


Exterior walls of homes


Bridges

Some silane coupling agents and their special features

KBM-5103

KBM-5103 is a silane coupling agent that contains acryloxy groups as the organic functional group. Compared to the methacryloxy group-based KBM-503, KBM-5103 has high radical reactivity and is more effective in UV and EB curing systems. Thanks to a more efficient reaction with the substrate, KBM-5103 promotes better adhesion and provides an improved reinforcing effect. It can also be used for modification of acrylic resins through copolymerization.

■ Chemical structure

$$(CH_3O)_3SiC_3H_6O\overset{}{\underset{\|\atop O}{C}}CH{=}CH_2$$

Features

■ Radical reactivity

To compare the reactivity of vinyl, acryloxy and methacryloxy groups, silicones containing these different functional groups were irradiated with an electron beam (EB) and their curing properties were compared. The silicone with acryloxy groups cured with a smaller EB dose than did those with methacryloxy or vinyl groups, which is indicative of its higher radical reactivity.

$$-(SiMe_2\text{-}O)_n\text{-}(SiMeR\text{-}O)_m\text{-}$$ R: 5 mol%

R (Functional group)	Minimum dose required for curing (kGy)
Vinyl	> 100
Methacryloxy	50
Acryloxy	20

■ Reinforcing effect

Using unsaturated polyester and glass fiber treated with KBM-5103, a laminate was prepared and tested in a comparison of flexural strength.
Compared to KBM-503, KBM-5103 provided at least a 10% greater strength improvement. (Glass content of laminates: 63–65%)

Treatment silane	Functional group	Flexural strength N/mm^2	Flexural strength ratio, %
Untreated	—	330	100
KBM-503	Methacryloxy	445	135
KBM-5103	Acryloxy	490	149

KBE-9007

KBE-9007 contains isocyanate groups as the organic functional group. Isocyanate groups react strongly with organic functional groups that contain active hydrogens, such as hydroxyl, amino and mercapto groups, and it is this reaction that allows for KBE-9007 to be used to introduce alkoxysilyl groups into urethane and other organic resins.

■ Chemical structure

$$(C_2H_5O)_3SiC_3H_6N{=}C{=}O$$

Application examples: Reaction with active hydrogens. The following examples involve systems which contain active hydrogens.

■ Preparation of an alkoxysilyl-functionalized crosslinking fluoropolymer solution

In simple steps, a one-component, room-temperature-cure coating agent can be prepared which offers outstanding adhesion, flexibility, impact resistance and solvent resistance.

Combine and mix the following ingredients for 2 hours under a nitrogen atmosphere: Solution of fluoropolymer which has hydroxyl groups in the side chains (50% xylene solution, OH number: 52 mgKOH/g, Viscosity: 5,000 mm^2/s at 50 °C): 15 kg, Xylene: 75 kg, KBE-9007: 8.6 kg (OH/NCO=1/1.5), Dibutyltin dilaurate: 0.0025 kg.

This yields a highly stable solution that shows no problems with flowability even after 20 days at 50 °C. The solution was applied to a chromate-treated aluminum sheet and cured at room temperature for 3 days. The film properties were then measured.

■ Film properties

Test item	Result
Xylene	200+ cycles
Cross-cut test	100/100
DuPont impact test	50 cm
T bending test	1T
Pencil hardness	4H

■ Improving adhesion of silicone rubber

KBE-9007 can be added to create an adhesive silicone compound.

First prepare what we will call “Compound A”, by combining the following ingredients in a kneader-mixer and mixing for 2 hours at 160 °C until homogenous: Methyl vinyl polysiloxane (viscosity: 5,000,000 mm^2/s): 100 parts, Hydrophobic fumed silica (specific surface area: 200 m^2/g): 40 parts, α, ω-dimethyl polysiloxane diol (viscosity: 20 mm^2/s): 2 parts.
Next, mix and knead the following ingredients using a 2-roll kneader: Compound A: 100 parts, KBE-9007: 1 part, 2,5-dimethyl-2,5-di(t-butylperoxy)hexane: 0.5 parts.

■ Adhesive strength

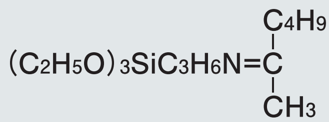
A sample adhesive was prepared as described above. The adhesive was applied to various substrates and then pressed in a die. Heat and pressure were applied, at 165 °C·20 kgf/cm^2 for 10 minutes.

Substrate	Adhesive strength, MPa
SUS304	0.83
SS40	0.86
Polyester board	1.01
Glass fiber-reinforced polyester board	0.74

KBE-9103

KBE-9103 is a silane coupling agent with a ketimine structure in which a primary amine is masked by a ketone compound. Ketimine structures react with moisture in the air or other moisture source and hydrolyze easily, releasing the ketone compound to become an active primary amine. KBE-9103 can be added to mixtures into which primary amines cannot be mixed as they are. KBE-9103 can be used in a wide range of applications including paints and adhesives.

■ Chemical structure



Features

■ Stability in epoxy resins

Compared to KBE-903, which contains a primary amine, KBE-9103 causes less thickening.

Product used	Viscosity mm^2/s			
	3 days	5 days	7 days	14 days
None	4.2	4.2	4.2	4.3
KBE-903	7.8	7.9	8.1	8.6
KBE-9103	4.4	4.4	4.5	4.7

Mixtures of 50 parts jER 827 (liquid epoxy resin mfd. by MITSUBISHI CHEMICAL CORPORATION), 50 parts toluene and 5 parts coupling agent were prepared and the change in viscosity over time was measured.

■ Adhesive effect

KBE-9103 provides an adhesive effect as good or better than that of KBE-903, the primary-amine containing product.

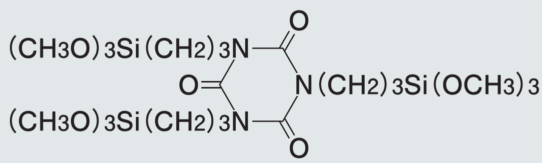
Substrate	Product used	Tensile strength MPa	
		Initial strength after 24 h	Water resistance test (95 °C/10 h)
Aluminum sheet	None	3.9	3.4
	KBE-903	6.1	5.2
	KBE-9103	7.6	6.4
Steel sheet	None	12.4	9.3
	KBE-903	15.7	13.5
	KBE-9103	17.8	14.1

Mixtures of 100 parts jER 827, 5 parts coupling agent and 10 parts curing agent (triethylenetetramine) were prepared and applied to the substrates to test adhesion.

KBM-9659

The molecules of KBM-9659 contain isocyanurate groups. This silane has good miscibility with organic resins, yet does not react strongly with organic resins, so it has good shelf-life even after compounding with organic resins. Because the alkoxy groups are polyfunctional, KBM-9659 can promote better adhesion to inorganic materials.

■ Chemical structure



Features

- ●Effectively improves adhesion of organic resins to inorganic materials.
- ●Low reactivity with organic resins, so provided that moisture is properly controlled, KBM-9659 will not harm the shelf-life of the resins.
- ●High boiling point, so it can be diluted directly with an organic solvent and used as a primer.

✻ KBM-9659 may crystallize during storage, but this does not affect product quality. If the product crystallizes, place the container in hot water (40–50 °C) and the product will soon return to a liquid state.

✻ KBM-9659 does not dissolve in water, and so cannot be used to prepare aqueous solutions.

■ General properties

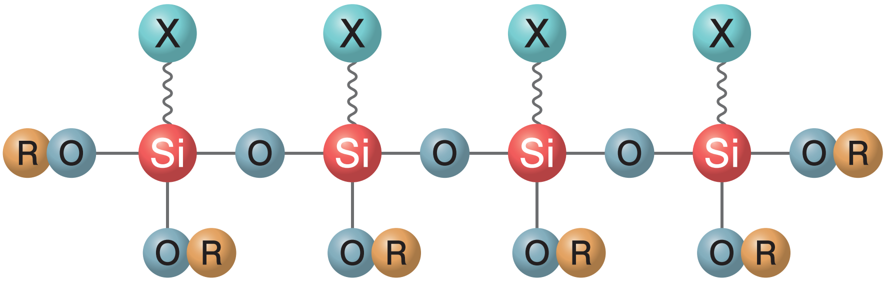
Item \ Product name		KBM-9659
Appearance		Pale yellow transparent
Viscosity 25 °C	mm^2/s	300
Specific gravity 25 °C		1.18
Refractive index 25 °C		1.458
Flash point	°C	186 (open cup)
Boiling point	°C	≥250
Active content	%	100

(Not specified values)

Alkoxy Oligomers

Alkoxy oligomers are resins of relatively low molecular weight which contain both organic groups and alkoxysilyl groups. Alkoxy oligomers that contain methyl and phenyl groups as the organic groups are common, while others contain epoxy groups, mercapto groups and other organic functional groups. These oligomers have many of the features of silane coupling agents. Alkoxy oligomers have advantages over silane monomers in that they are low volatility, produce fewer alcohol byproducts, and are polyfunctional.

■ Structural diagram



■ General properties

Product name	Organic substituent groups	Alkoxy groups	Viscosity 25°C mm²/s	Refractive index 25°C	Alkoxy group content, wt·%	SiO_2 content wt·%	Features	UN hazard classification
X-41-1053	Epoxy	Methoxy/Ethoxy	12	1.414	50	39	Epoxy equivalent 830 g/mol	Not applicable
X-41-1059A	Epoxy	Methoxy/Ethoxy	30	1.434	42	35	Epoxy equivalent 350 g/mol	Not applicable
X-41-1056	Methyl/Epoxy	Methoxy	40	1.442	27	41	Epoxy equivalent 280 g/mol	Not applicable
X-41-1805	Mercapto	Methoxy/Ethoxy	30	1.418	50	43	Mercapto equivalent 800 g/mol	Not applicable
X-41-1818	Mercapto	Ethoxy	14	1.417	60	42	Mercapto equivalent 850 g/mol	UN-1993
X-41-1810	Methyl/Mercapto	Methoxy	5	1.422	30	53	Mercapto equivalent 450 g/mol	Not applicable
X-40-2651	Methyl/Amine	Methoxy	20	1.405	7	75	Forms rubber film	UN-1993
X-40-2655A	Methyl/Methacryloxy	Methoxy	16	1.440	26	36	Methacrylate equivalent 280 g/mol	Not applicable
KR-513	Methyl/Acryloxy	Methoxy	35	1.450	20	30	Acrylate equivalent 210 g/mol	Not applicable
KC-89S	Methyl	Methoxy	5	1.394	45	59	Low polymer	Not applicable
KR-500	Methyl	Methoxy	25	1.403	28	63	Medium polymer	Not applicable
X-40-9225	Methyl	Methoxy	100	1.407	24	67	High polymer	Not applicable
X-40-9246	Methyl	Methoxy	80	1.407	12	72	Improves flexibility	UN-1993
X-40-9250	Methyl	Methoxy	160	1.407	25	73	Improves flexibility, forms thick films	Not applicable
KR-401N	Methyl/Phenyl	Methoxy	20	1.432	33	56	Low phenyl content, curable	UN-1993
X-40-9227	Methyl/Phenyl	Methoxy	15	1.460	15	59	Improves softness	Not applicable
X-40-9247	Methyl/Phenyl	Methoxy	100	1.508	16	46	High hardness, low volatile content	Not applicable
KR-510	Methyl/Phenyl	Methoxy	100	1.509	17	44	High hardness	Not applicable
KR-9218	Methyl/Phenyl	Methoxy	40	1.529	15	40	Forms med. hardness film	Not applicable
KR-213	Methyl/Phenyl	Methoxy	16	1.525	20	38	High phenyl content	Not applicable
X-40-2308	—	Methoxy	4	1.387	65	51	No organic substituent groups, high hardness	Not applicable
X-40-9238	—	Ethoxy	13	1.402	70	45	No organic substituent groups, high hardness	Not applicable

(Not specified values)

Catalysts

If the alkoxy oligomer is to be used as a coating compound or for modification of organic resins, it must be used in combination with a catalyst. Commonly used catalysts include products based on organometallic compounds, acids and amine compounds. The following are particularly suitable as curing and reaction catalysts.

■ Catalysts for alkoxy oligomers

Product name	Type	Active ingredient, %	Metal content, %	Features	Amount to add (guide), %
D-20	Titanium	100	21	General purpose catalyst with mild reactivity	2-5
D-25	Titanium	100	14	Higher activity than D-20	0.5-3
DX-9740	Aluminum	100	9	Somewhat slow to dry, but forms hard films	3-10
X-40-2309A	Phosphate	100 (contains reactive diluent)	—	Ultra fast-cure type with good drying characteristics	10-50

Catalysts' effects on film characteristics

Methylated oligomers have excellent hydrolytic properties, and when paired with the right curing catalysts, these oligomers can be used as room-temperature/moisture-cure coating agents that form cured films which are very hard and have excellent abrasion resistance. Methylated oligomers are thus ideally suited for use as a protective coating for floors and for exterior painting applications.
Titanium based curing catalysts are frequently used. A variety of methylated oligomers have been developed, with molecular weights ranging from low to high and with enhanced flexibility (crack resistance) and with thick-film properties. By pairing these oligomers with the proper catalyst, it should be possible to achieve the desired film characteristics. Sample pairings of methylated oligomers and curing catalysts are shown below. Generally speaking, it is possible to obtain hard films that cure relatively quickly by using a low-molecular-weight oligomer or by increasing the amount of curing catalyst. The downside is that there will be a higher crosslinking density when the film forms, which means that bending and impact forces will be more likely to crack the film. Therefore, for thick-film applications, or to improve crack resistance, it is best to use a methylated oligomer with a high molecular weight or one designed for enhanced flexibility.

■ Differences in film characteristics obtained with different methylated oligomer/curing catalyst pairings

Alkoxy oligomer	Curing catalyst (amount added, %)	Film thickness (µm)	Tack-free time	Pencil hardness	Bending/impact resistance
KR-500	D-20 (2)	25	40	H	△
KR-500	D-20 (4)	25	25	2H	△–×
KR-500	DX-9740 (5)	25	100	5H	×
X-40-9225	D-20 (3)	30	60	H	○
KR-500/X-40-9250 (=80/20)	D-20 (2)	80	75	F	○

○: Good △: Fair ×: Poor
✻Substrate: polished steel sheet, Curing conditions: 25 °C/70%RH/7 days (Tack-free time varies depending on temperature & humidity.)

Formulation examples

Alkoxy oligomers can be used in various combinations to obtain cured films with the desired characteristics.

■ Use of alkoxy oligomers in paints

Alkoxy oligomer/catalyst composition	Time to cracking (h)		Film yellowing
	Without primer	With primer	
KR-500/X-40-9225/D-20 (=80/20/5)	100	100	◎
X-40-9246/X-40-9225/X-40-2309A (=50/20/30)	400	500	◎
KR-510/X-40-2309A (=70/30)	1,000	1,000	○–◎
(Non-silicone material)	—	1,000	×

◎: No yellowing ○: Minimal yellowing ×: Significant yellowing
✻ Measuring device: EYE SUPER UV TESTER (SUV-W11 tester with metal halide lamp, manufactured by Iwasaki Electric)
Test conditions: in accordance with JTM G 01; 4 hour irradiation (luminous intensity: 100 mW/cm^2) followed by 2 hour darkness/condensation = 1 cycle
Black panel temp./humidity: during irradiation: 63±3 °C/50±10%RH, during condensation: 30±3 °C/≥95%RH
Substrate: straight sheet (acrylic, with and without primer), Film thickness: 30-50 µm, Curing conditions: 25 °C/70%RH × 7 days

Silanes

Shin-Etsu's silane products are a group of organosilicon compounds comprised of alkoxy silanes and silazanes.
Silanes have many applications in a wide variety of fields.
They are commonly applied to the surface of inorganic substrates to improve water repellency,
added to inorganic fillers to improve their dispersibility in organic polymers,
and used for surface modification of inorganic materials.

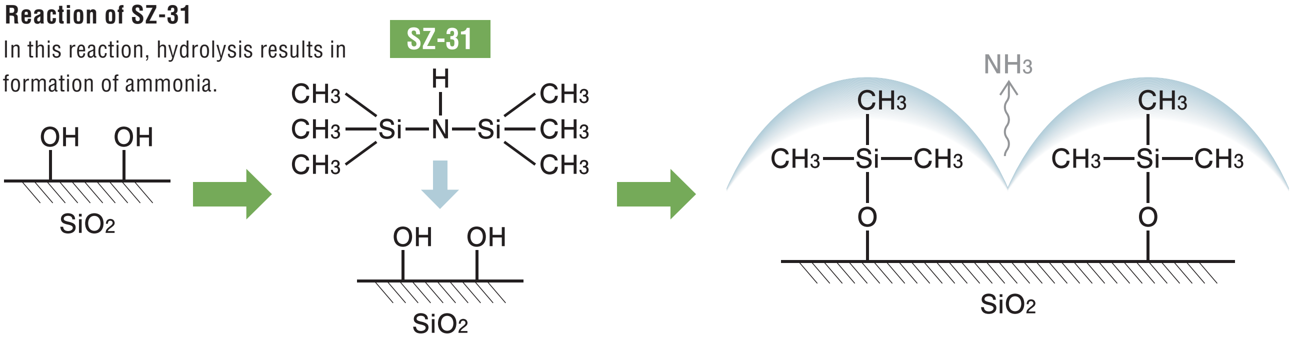
General properties

	Product name	Chemical name	Structural formula	Molecular weight	Specific gravity 25°C	Refractive index 25°C	Boiling point °C	Flash point °C	Minimum covering area, m^2/g	UN hazard classifi-cation	Existing substances No.	CAS No.
Alkoxy silane	**KBM-13 (LS-530)**	Methyltrimethoxysilane	$(CH_3O)_3SiCH_3$	136.2	0.95	1.369	102	8*1	573	UN-1993	2-2052	1185-55-3
	KBM-22 (LS-520)	Dimethyldimethoxysilane	$(CH_3O)_2Si(CH_3)_2$	120.2	0.86	1.371	82	-10	649	UN-1993	2-2052	1112-39-6
	KBM-103 (LS-2750)	Phenyltrimethoxysilane	$(CH_3O)_3SiC_6H_5$	198.3	1.06	1.473	218	94*2	393	Not applicable	3-2635	2996-92-1
	KBE-13 (LS-1890)	Methyltriethoxysilane	$(C_2H_5O)_3SiCH_3$	178.3	0.89	1.383	143	40*1	437	UN-1993	2-2052	2031-67-6
	KBE-22 (LS-1370)	Dimethyldiethoxysilane	$(C_2H_5O)_2Si(CH_3)_2$	148.3	0.83	1.384	114	15*1	526	UN-2380	2-2052	78-62-6
	KBE-103 (LS-4480)	Phenyltriethoxysilane	$(C_2H_5O)_3SiC_6H_5$	240.4	0.99	1.459	236	111*2	324	Not applicable	3-2635	780-69-8
	KBM-3063 (LS-3130)	Hexyltrimethoxysilane	$(CH_3O)_3Si(CH_2)_5CH_3$	206.4	0.91	1.406	202	81*2	378	Not applicable	2-2052	3069-19-0
	KBE-3063 (LS-4808)	Hexyltriethoxysilane	$(C_2H_5O)_3Si(CH_2)_5CH_3$	248.4	0.88	1.408	120.6*3	95*2	314	Not applicable	2-2052	18166-37-5
	KBM-3103 (LS-5258)	Decyltrimethoxysilane	$(CH_3O)_3Si(CH_2)_9CH_3$	262.5	0.90	1.421	132*4	135*2	297	Not applicable	2-3512	5575-48-4
	KBM-7103 (LS-1090)	Trifluoropropyl-trimethoxysilane	$(CH_3O)_3SiCH_2CH_2CF_3$	218.2	1.14	1.352	144	23*1	357	UN-1993	2-2079	429-60-7
Silazane	**SZ-31 (LS-7150)**	Hexamethyldisilazane	$(CH_3)_3SiNHSi(CH_3)_3$	161.4	0.77	1.408 (20°C)	126	14*1	967	UN-3286	2-2955 or 2-2044	999-97-3
Siloxane	**KPN-3504**	Siloxane with hydrolyzable groups	Proprietary	—	0.97	1.405	—	108	—	Not applicable	Registered	—

*1: Closed cup *2: Open cup *3: 21 mmHg (2.8 kPa) *4: 10 mmHg (1.3 kPa) (Not specified values)
*( ) Sold as reagent under names in parentheses. Amounts under 1 kg are reagents.
*1 kPa: 7.5 mmHg

Reaction of SZ-31

In this reaction, hydrolysis results in formation of ammonia.



Water repellency (surface properties)

1. Water repellency (on glass substrate)

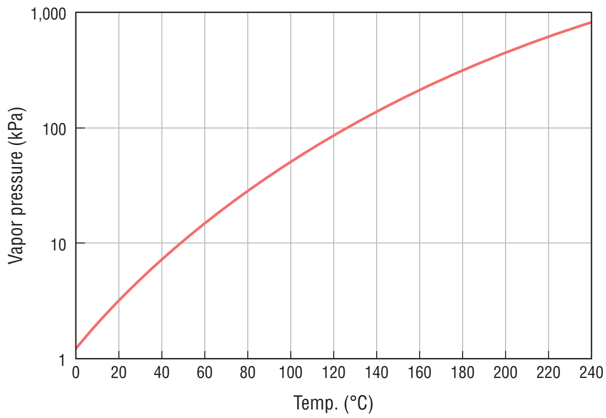
Silane	Water contact angle (°)
KBM-13	63
SZ-31	66
KBM-3103	84

2. Surface energy reduction
Critical surface tension of silane treated surfaces (γ crit)

Silane	γ crit (mN/m)
KBM-7103	20.6
KBM-13	22.5
KBM-103	40.0

Vapor pressure curve of SZ-31

Vapor pressure curve of SZ-31



Silylating Agents

Silylating agents are used in reactions to introduce organosilyl groups into inorganic and organic materials.
Active hydrogens in the molecules are substituted by silyl groups,
thereby forming protecting groups for the active hydrogens.
Silylating agents can also improve the solubility of the molecules in non-polar solvents
and improve heat resistance or to increase volatility.

■ General properties

Product name	Chemical name	Structural formula	Boiling point °C	Flash point °C	UN hazard classification	Existing substances No.	CAS No.
KA-31 (LS-260)	Trimethylsilyl chloride	$(CH_3)_3SiCl$	57	-15	UN-1298	2-2041	75-77-4
SZ-31 (LS-7150)	Hexamethyldisilazane	$(CH_3)_3SiNHSi(CH_3)_3$	126	14	UN-3286	2-2955 or 2-2044	999-97-3
BTSU (LS-7180)	N,N'-Bis(trimethylsilyl)urea	$(CH_3)_3SiNHCONHSi(CH_3)_3$	—	—	UN-1325	2-3250	18297-63-7
BSTFA (LS-7240)	N,O-Bis(trimethylsilyl)trifluoroacetamide	$(CH_3)_3SiOC(CF_3)=NSi(CH_3)_3$	46°C/2.27 kPa	34	UN-1993	2-3613	25561-30-2
TMST (LS-415)	Trimethylsilyl trifluoromethanesulfonate	$(CH_3)_3SiOSO_2CF_3$	140	40	UN-2920	2-3617	27607-77-8
TESC (LS-1210)	Triethylsilyl chloride	$(C_2H_5)_3SiCl$	145	39	UN-2986	2-2041	994-30-9
TBMS (LS-1190)	t-Butyldimethylsilyl chloride	tert-Bu$(CH_3)_2SiCl$	125	28	UN-2925	2-2041	18162-48-6
TIPSC	Triisopropylsilyl chloride	i-Pr_3SiCl	59°C/1.07 kPa	63	UN-2987	2-2041	13154-24-0
CIPS (LS-7612)	1,3-Dichloro-1,1,3,3-tetraisopropyldisiloxane	Cl(i-Pr)$_2$SiOSi(i-Pr)$_2$Cl	108°C/0.53 kPa	110	UN-2987	2-3942	69304-37-6
CMTMS (LS-460)	Chloromethyltrimethylsilane	$ClCH_2Si(CH_3)_3$	97	< -2	UN-1993	2-3846	2344-80-1
TES (LS-1320)	Triethylsilane	$(C_2H_5)_3SiH$	107	-1	UN-1993	Small quantity exemption	617-86-7
HBS	t-Butyldimethylsilane	tert-Bu$(CH_3)_2SiH$	86	-14	UN-1993	2-3696	29681-57-0
TMSA (LS-610)	Trimethylsilylacetylene	$HC\equiv CSi(CH_3)_3$	53	-34	UN-1993	2-3779	1066-54-2
PMDS (LS-7120)	Hexamethyldisilane	$(CH_3)_3SiSi(CH_3)_3$	112	-9	UN-1993	2-3711	1450-14-2
ATMS (LS-1100)	Allyltrimethylsilane	$CH=CHCH_2Si(CH_3)_3$	86	7	UN-1993	2-3737	762-72-1
TMVS (LS-720)	Trimethylvinylsilane	$CH_2=CHSi(CH_3)_3$	54	-34	UN-1993	2-4007	754-05-2

✻1 kPa: 7.5 mmHg (Not specified values)

■ Reactivity of active hydrogens and silylating agents

Generally speaking, hydroxyl groups are the most reactive, followed by phenol groups, carboxyl groups, amino groups and mercapto groups, but this order may change depending on the type of silylating agent used as well as the reaction solvent and type and amount of catalyst.

$$ROH > ArOH > -CO_2H > -NH > -CONH > -SH$$

■ Stability of silylated compounds

Through hydrolysis, silylated compounds can be returned to the original active hydrogen compounds. Hydroxyl groups have the greatest hydrolytic stability, followed by mercapto groups, phenol groups, carboxyl groups and amino groups. Yet even the most stable hydroxyl groups can be easily hydrolyzed by hydrochloric acid. Hydrolytic resistance can be improved by using silylating agents with bulky alkyl groups.

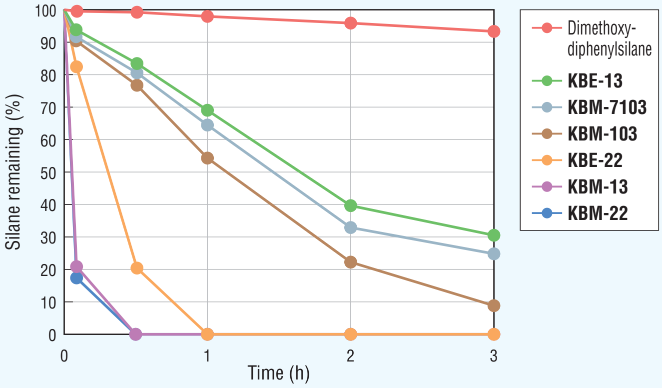
$$-O(TMS) > -S(TMS) > ArO(TMS) > -CO_2(TMS) > -N(TMS)$$

✻TMS: trimethylsilyl groups

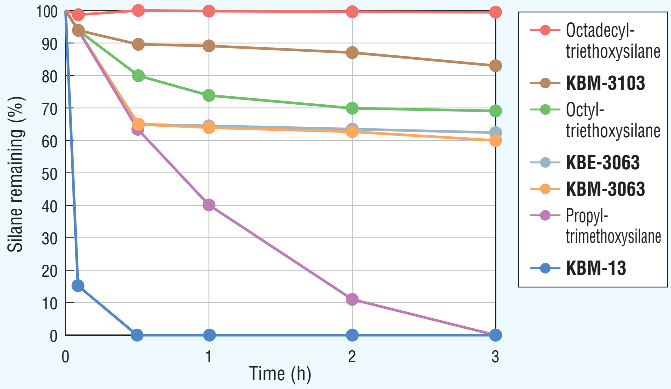
Hydrolytic properties

Hydrolysis rates of silanes

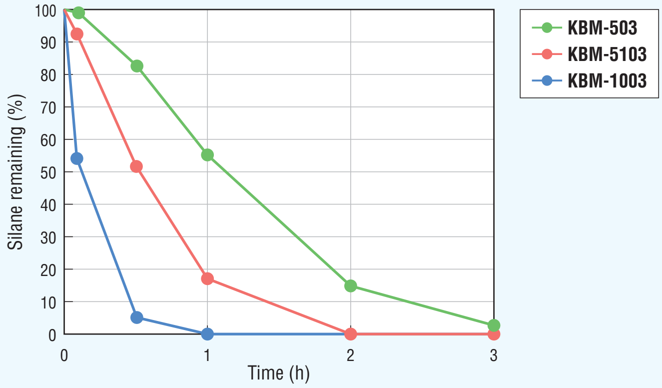
Hydrolytic properties of different functional groups



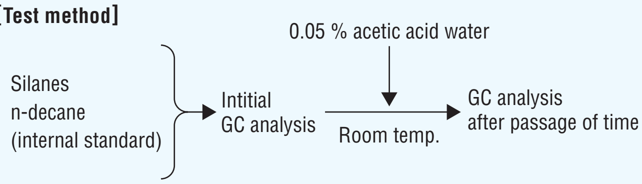
Hydrolytic properties of long-chain alkyl silanes



Hydrolytic properties of different polymerizable groups (polymerizable silanes)



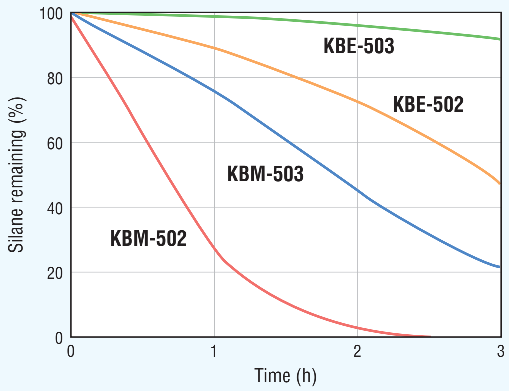
[Test method]



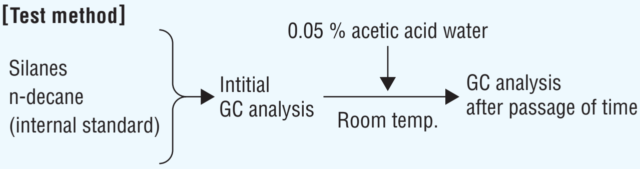
1. Silanes and n-decane were mixed.
2. Gas chromatography (GC) was performed on the mixed liquids and the initial residual amounts were determined.
3. 0.05% acetic acid water was added, and the liquids were agitated at room temperature.
4. GC was performed again later and the residual rates were calculated based on the initial residue amounts.

Numbers of methoxy & ethoxy functional groups and their effect on hydrolytic properties

The graph below presents data for methacryloxy silanes, showing how the type and number of hydrolyzable functional groups (methoxy & ethoxy) affect the hydrolysis rate.
When the silanes were pH-adjusted with acetic acid water and hydrolyzed, KBM-502 hydrolyzed fastest, followed by KBM-503, KBE-502, and KBE-503.

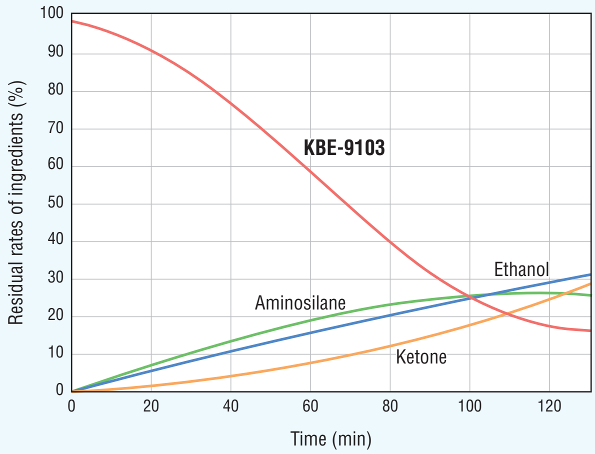


[Test method]



Hydrolysis rate of KBE-9103

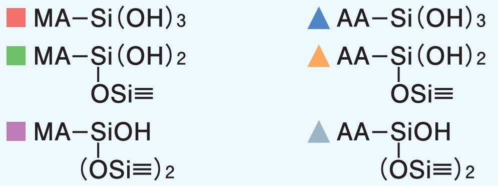
When KBE-9103 comes in contact with water, the ketimines and alkoxysilyl groups hydrolyze, forming amino and silanol groups.
The speed of hydrolysis will be about the same under these conditions.

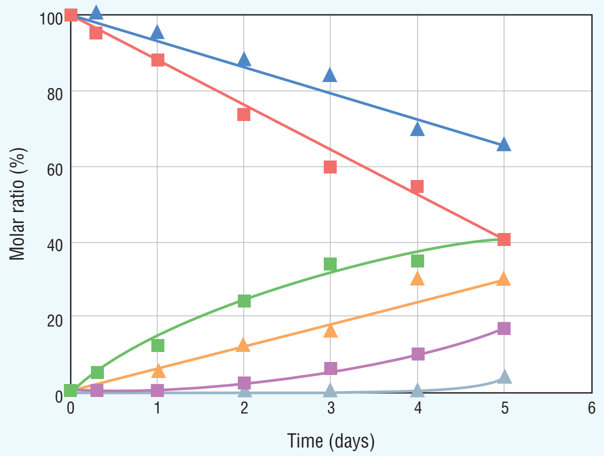


Test conditions: Room temperature, blend/ silane:water=5:1
Analysis method: Gas chromatography (internal standard THF)

Comparison of condensation reaction properties of methacryloxy silane and acryloxy silane

In comparing a methacryloxy silane (KBM-503) with an acryloxy silane (KBM-5103), it was found that condensation proceeds more slowly for the acryloxy silane.

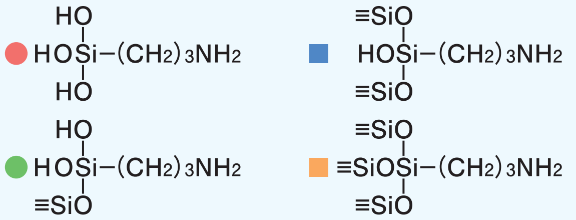




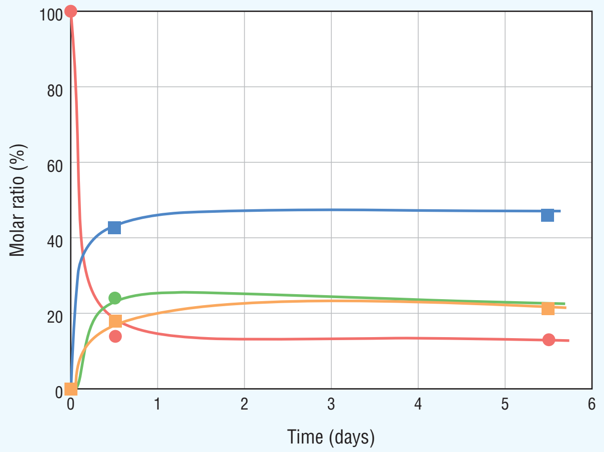
Conditions: 2% silane, 0.3% acetic acid, 50% ethanol, 48% water
Methacryloxy silane: **KBM-503** MA: $CH_2{=}CCH_3COO(CH_2)_3$—
Acryloxy silane: **KBM-5103** AA: $CH_2{=}CHCOO(CH_2)_3$—

Condensation behavior of amino silane in aqueous solution

An amino silane (KBE-903) was found to be very stable in aqueous solutions.



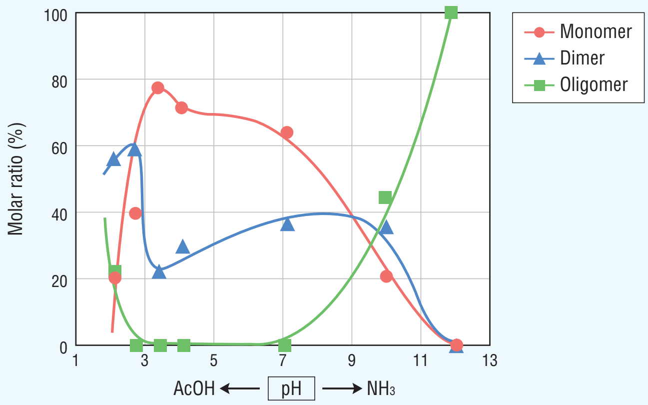
KBE-903 5% aqueous solution (pH=11.1)



Stability of epoxy silane-water solutions

The stability of a silane-water solution will vary greatly as the pH of the solution changes. It also varies depending on the concentration. The graphs below show the effects of pH and concentration on the stability of an epoxy silane (KMB-403).

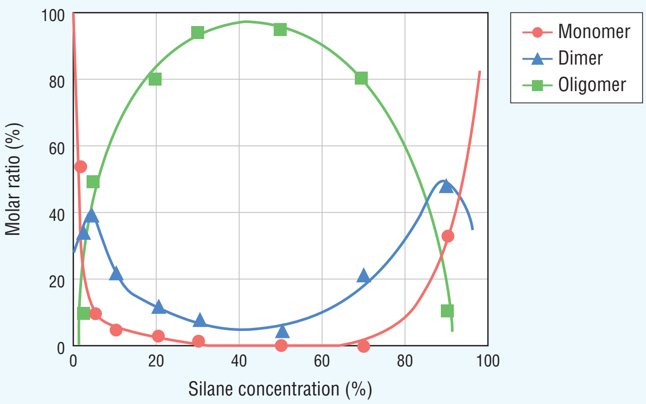
Epoxy silane-water solution: Stability vs. pH



Conditions: 10% silane, 30 °C, 4 hours

Silane: $(CH_3O)_3SiC_3H_6OCH_2CH{-}CH_2$ (epoxide O bridging CH–CH2)

Epoxy silane-water solution: Stability vs. concentration



Conditions: 1% acetic acid (pH=3), 30 °C, 48 hours

Silane: $(CH_3O)_3SiC_3H_6OCH_2CH{-}CH_2$ (epoxide O bridging CH–CH2)

Solubility in water

The alkoxysilyl groups in a silane coupling agent react with in water to form silanol groups.
These silanol groups are unstable and over time will undergo condensation. This results in formation of siloxane linkages, and ultimately gelation.
Silanol groups are generally unstable in aqueous solutions, but their stability improves if the solution is mildly acidic.
Meanwhile, amino silanes are very stable in aqueous solutions, due to interaction of the amino groups.
Methods for improving a solution's shelf-life include adjusting the pH of the liquid (pH 4–5), combining it with alcohol, and storing it at room temperature or below.

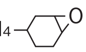
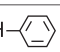
■ Solubility and stability at optimum pH

Product name	Solubility (pH of aqueous solution)	Shelf-life
KBM-1003	○ (3.9)	Up to 10 days
KBE-1003	○ (3.9)	Up to 10 days
KBM-303	○ (4.0)	Up to 30 days
KBM-403	◎ (5.3)	Up to 30 days
KBE-402	○ (4.0)	Up to 10 days
KBE-403	○ (4.0)	Up to 10 days
KBM-1403	Insoluble	—
KBM-502	○ (4.0)	Up to 1 day
KBM-503	○ (4.2)	Up to 1 day
KBM-5103	○ (4.2)	Up to 3 days
KBM-602	◎ (10.0)	Up to 30 days
KBM-603	◎ (10.0)	Up to 30 days
KBM-903	◎ (10.0)	Up to 30 days
KBE-903	◎ (10.0)	Up to 30 days
KBM-573	○ (4.0)	Up to 1 day
KBM-803	○ (4.0)	Up to 1 day
KBE-846	Insoluble	—

✻ Solubility
◎: 1% silane-water solution can be prepared without adjusting pH of aqueous solution
○: 1% silane-water solution can be prepared if pH of aqueous solution is adjusted
Insoluble: silane-water solution cannot be prepared

✻ Information on shelf-life should be taken as a guide.
Shelf-life will vary depending on usage conditions and intended use.

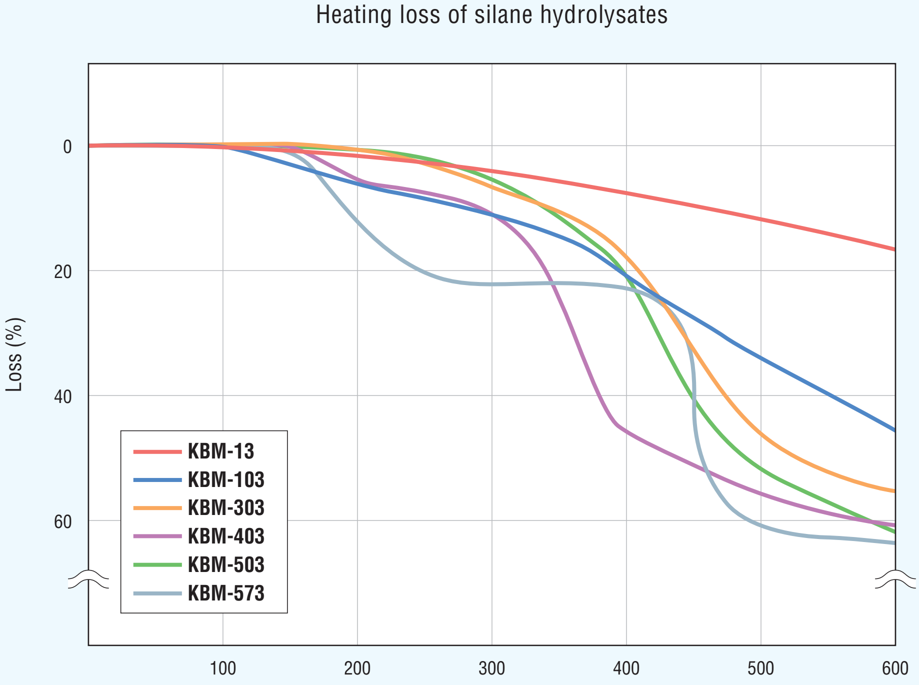
Solubility parameters of silane coupling agents

Product name	Structural formula	(RO)3Si-R-Y	-R-Y
KBM-1003	$(CH_3O)_3SiCH=CH_2$	7.49	7.00
KBE-1003	$(C_2H_5O)_3SiCH=CH_2$	7.76	7.00
KBM-303	$(CH_3O)_3SiC_2H_4$– 	8.58	9.38
KBM-403	$(CH_3O)_3SiC_3H_6OCH_2\overset{O}{\overbrace{CH-CH_2}}$	8.49	9.29
KBE-403	$(C_2H_5O)_3SiC_3H_6OCH_2\overset{O}{\overbrace{CH-CH_2}}$	8.51	9.29
KBM-502	$(CH_3O)_2\overset{CH_3}{Si}C_3H_6O\underset{O}{C}\overset{CH_3}{C}=CH_2$	8.53	9.48
KBM-503	$(CH_3O)_3SiC_3H_6O\underset{O}{C}\overset{CH_3}{C}=CH_2$	8.66	9.48
KBM-603	$(CH_3O)_3SiC_3H_6NHC_2H_4NH_2$	9.00	10.24
KBM-803	$(CH_3O)_3SiC_3H_6SH$	8.49	9.57
KBM-903	$(CH_3O)_3SiC_3H_6NH_2$	8.56	9.86
KBE-903	$(C_2H_5O)_3SiC_3H_6NH_2$	8.56	9.86
KBM-573	$(CH_3O)_3SiC_3H_6NH$– 	9.15	10.30
KBE-9007	$(C_2H_5O)_3SiC_3H_6N=C=O$	9.17	11.14

✻ Calculated from energy of evaporation and molar volume as determined by the Fedor's method.

Heating loss of silane hydrolysates

Measured in a heated state.

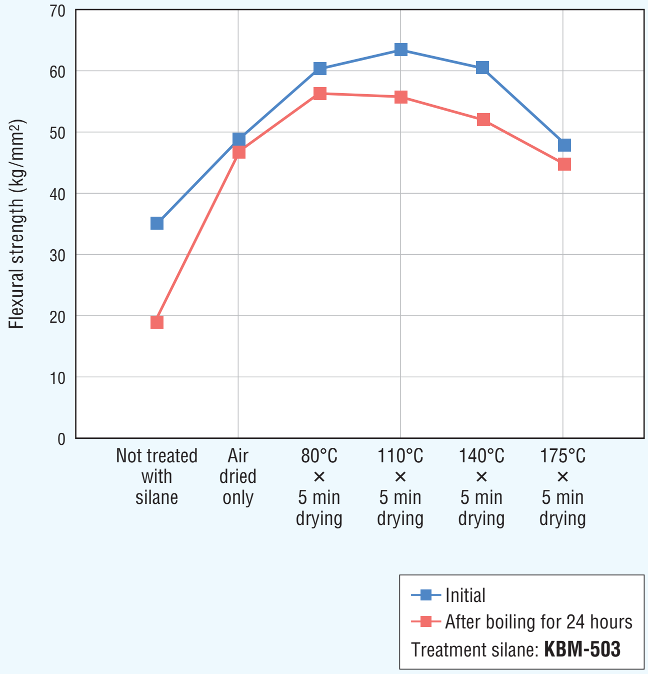


✻ In air, rate of temperature increase: 7.5 °C/min

Change in performance caused by dehydration condensation reaction

Comparison of treatment of polyester laminates

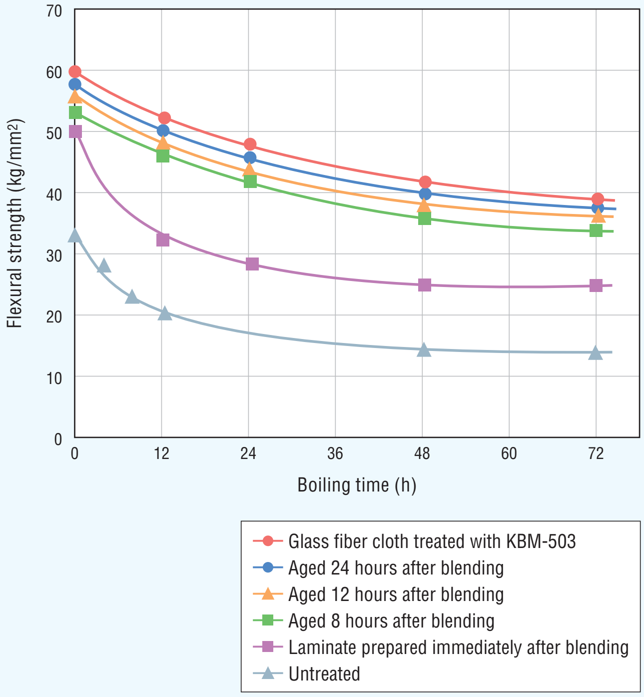
We compared the effects of different drying conditions on effectiveness of treatment. It was found that drying the silane coupling agent for around 5 minutes at 110 °C after application gave the best results.



Effects of aging on organic resin blends

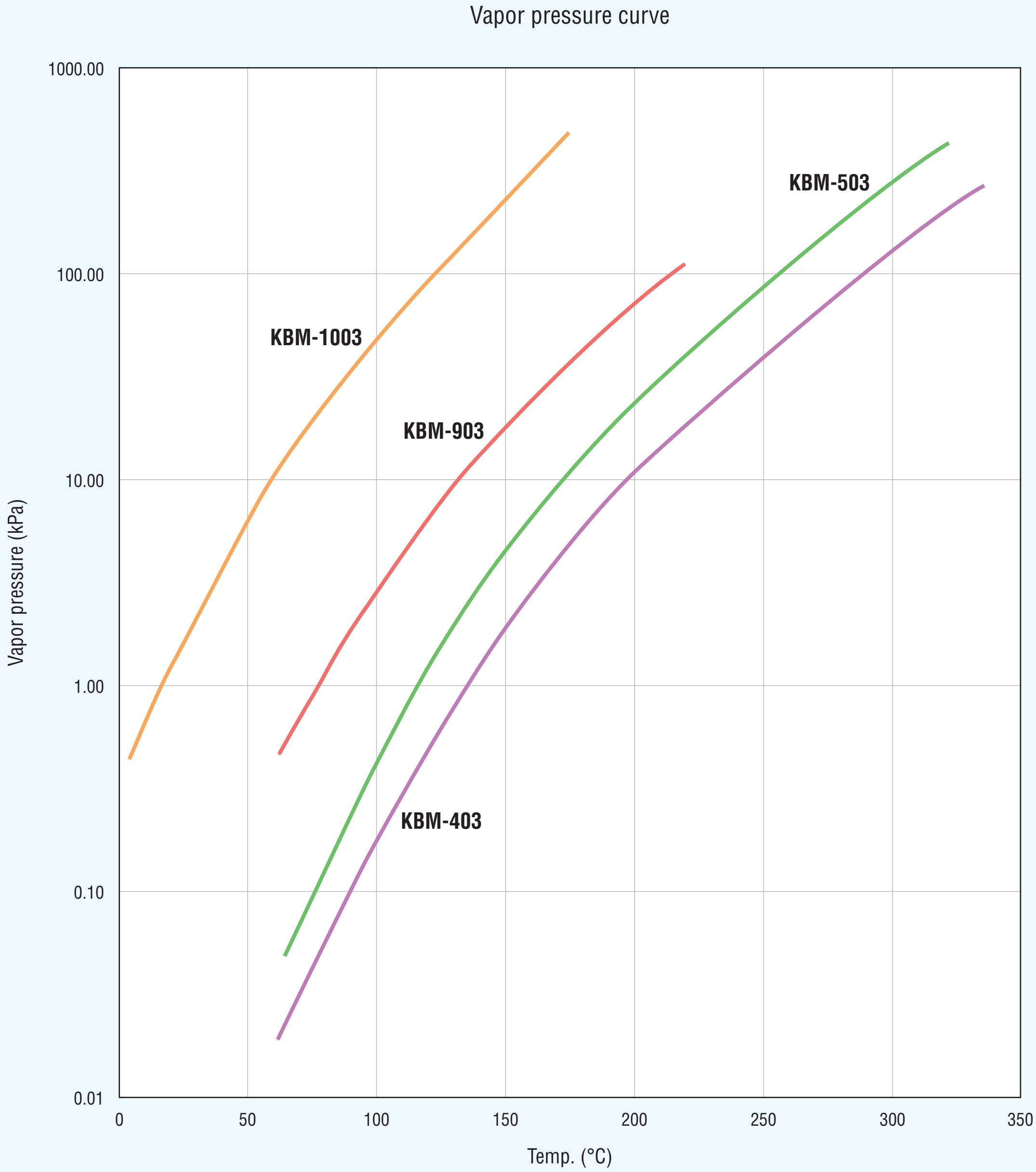
Application to polyester resin

When coupling agents are added via the integral blending method and aged at room temperature, the coupling agent migrates to the interface with the inorganic material. The effect is close to that achieved with pretreated glass fiber cloth.



Vapor pressure curve

Most silane coupling agents are compounds that have boiling points, and have vapor pressures which are unique to each compound.
The graph below shows the relationship between vapor pressure and temperature for some typical silane coupling agents.



Treating inorganic fillers with alkoxy silanes or silazanes: treatment amounts and evaluation

Treatment amount

The amount of treatment used for fillers is normally 0.5–2% by weight. The model equation here can be used as a guide with respect to the amount of silane required to surface-treat fillers to produce a monomolecular film on the filler particles.

$$\text{Silane treatment amount (g)} = \frac{\text{Weight of filler (g)} \times \text{Specific surface area of filler (m}^2\text{/g)}}{\text{Minimum covering area of the silane (m}^2\text{/g)}}$$

Evaluation

29Si NMR is a useful technique for determining how effectively a filler has been treated with a silane. For fillers that have been given a hydrophobic treatment, a simple method is to look at their hydrophobicity (methanol wettability) as an indicator of the degree of treatment, in which case higher hydrophobicity indicates a higher degree of treatment.

Checking the results of hydrophobic treatment

(1) Weigh out 0.5 g of the sample into a 500 ml Erlenmeyer flask.
(2) Add 50 ml of ion-exchange softened water to (1) and agitate with a magnetic stirrer.
(3) While continuing agitation, drip in methanol using a burette. When all of the sample is in suspension in the softened water, note the amount of methanol that has been dripped in.
(4) Determine hydrophobicity using the following equation.

$$\text{Hydrophobicity} = \frac{\text{Methanol drip amount (ml)} \times 100}{\text{Methanol drip amount (ml)} + \text{Ion-exchange softened water amount (ml)}}$$

Cleaning silane from reactors, containers, pipes, etc.

The following methods are good for cleaning, but keep in mind that results will vary depending on the type of silane (hydrophilic, hydrophobic), the material being cleaned (glass, metal, plastic), whether the silane has simply adhered to or has reacted onto the surface, and how much has built up. Use proper caution when handling solvents and alkalis.

1. Cleaning with solvents

This method involves cleaning off the silane by placing equipment in an organic solvent (alcohol, aromatic solvent, etc.). Agitation and heating will yield better cleaning results. With some physical effort, such as scrubbing with a brush, the results will be even better. Clean the insides of pipes by flushing them with large amounts of solvent.

2. Cleaning with alkalis

If the silane has reacted onto to the surface or has built up in significant amounts, cleaning with a solvent will not be sufficient. If so, the silane can be removed by placing equipment in an alkaline water solution (e.g. 50% potassium hydroxide-water solution). Again, agitation and heating will yield better cleaning results. When cleaning stainless steel, the solution can be heated to around 80 °C without problems. However, glass-lined equipment will be damaged at this temperature, so such equipment should not be soaked more than a few hours at temperatures not higher than around 50 °C. After cleaning, be sure to remove the alkaline component by washing thoroughly with water or alcohol.

Packaging

For inquiries about packaging, please contact one of our sales departments around the world (see listing on final page).

Handling precautions

Product quality, storage and handling

1. Store in a cool, dark place (out of direct sunlight, at room temperature or below) and take care to prevent exposure to moisture.
2. When exposed to water or moisture, silane coupling agents undergo hydrolysis and degrade, and in the process will release substances which include methanol, ethanol and hydrogen chloride. Do not leave product containers open, and always close tightly after use to prevent water and moisture from entering the container. Ideally, when closing containers, the air in the container should be replaced with dry nitrogen.
3. If you need a special high purity product for use in electronics materials manufacturing or other application, please discuss your needs with a Shin-Etsu sales representative.
4. Be sure to read the Material Safety Data Sheets (MSDS) for these products before use. MSDS are available from the Shin-Etsu Sales Department.
5. Contact the Shin-Etsu Sales Department to discuss issues concerning the export of these products.

Safety & hygiene

1. Ensure there is proper ventilation when using these products. Avoid breathing of vapors from products or their hydrolysis products, and avoid bodily contact.
2. Wear rubber gloves, safety glasses and other protective gear to prevent contact with the skin and mucous membranes. In case of contact, wash immediately and thoroughly with running water.
3. In case of eye contact, immediately flush eyes with plenty of running water, and consult a physician if necessary.
4. If products get on clothing, wash off with running water.
5. Be sure to wash hands thoroughly after handling products and before eating, drinking or smoking.
6. In case of spills, wash with plenty of water or soak up the spilled liquid using rags or sand and dispose of it by incineration.

Additional information

Shin-Etsu has pages devoted to our silane compounds on our website. Through the website, you can inquire about specific products, request samples, and download catalogs online.

http://www.silicone.jp/

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