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ALPHA-SILANE-TERMINATED POLYMERS AS NOVEL  
BINDERS FOR FAST CURING ELASTIC ADHESIVES  
PRESENTATION BY WOLFRAM SCHINDLER AT  
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CREATING TOMORROW'S SOLUTIONS



# ALPHA-SILANE-TERMINATED POLYMERS AS NOVEL BINDERS FOR FAST-CURING ELASTIC ADHESIVES

Text: Dr. Wolfram Schindler

Where bonded joints are subjected to dynamic loads, adhesives are needed which cure to form a strong, yet elastic material. Recently,  $\alpha$ -silane-terminated polyethers have become available as binders for elastic adhesives. These hybrid polymers, a completely new development from WACKER SILICONES, impart high tensile strength combined with good and adjustable elasticity. The new binders are notable for their low viscosity, are easy to process, crosslink extremely quickly and are not known to be harmful in any way.

There are many applications in which the bonded joint must be not only strong but also elastic. Elasticity becomes more important as soon as the bonded joint is subjected to a dynamic load. Since elastic adhesives deform reversibly and can absorb dynamic loads in the process, they have become firmly established in many applications (**Figures 1a and 1b**).

It would be unthinkable today to assemble the bodywork of buses, railroad cars, campers, mobile homes and refrigerated trucks, for example, without elastic adhesives. Elastic adhesives are also becoming increasingly common in aircraft construction and shipbuilding.

The binders used in elastic adhesives derive mainly from two polymer classes, namely polyurethanes (PUR) and silane-terminated polymers. Polyurethanes crosslink using isocyanate chemistry. Silane-terminated polymers crosslink via the alkoxysilane units attached to the ends of the polymer chain. These polymers form highly stable silicon-oxygen-silicon-bonds when they cure, which is typical of silicones.

Aside from undeniable advantages, all the commercially available binders suitable for formulating elastic adhesives also have some specific disadvantages. Polyurethane systems, for example, cure to produce materials with very high tensile strengths. However, in terms of their elongation at break and adhesion to various substrates (without pretreatment), they are inferior to the vulcanizates of adhesives formulated with silane-terminated polyethers. Silane-terminated polyurethanes, which could be described as a combination of the two systems, are either not very easy to compound because of their high viscosity, or their elongation at break is very low.



**Figures 1a and 1b: Bonding a car windshield in place. The elastic adhesive also helps to make the car body more rigid and simultaneously serves as a seal to keep out rainwater.**

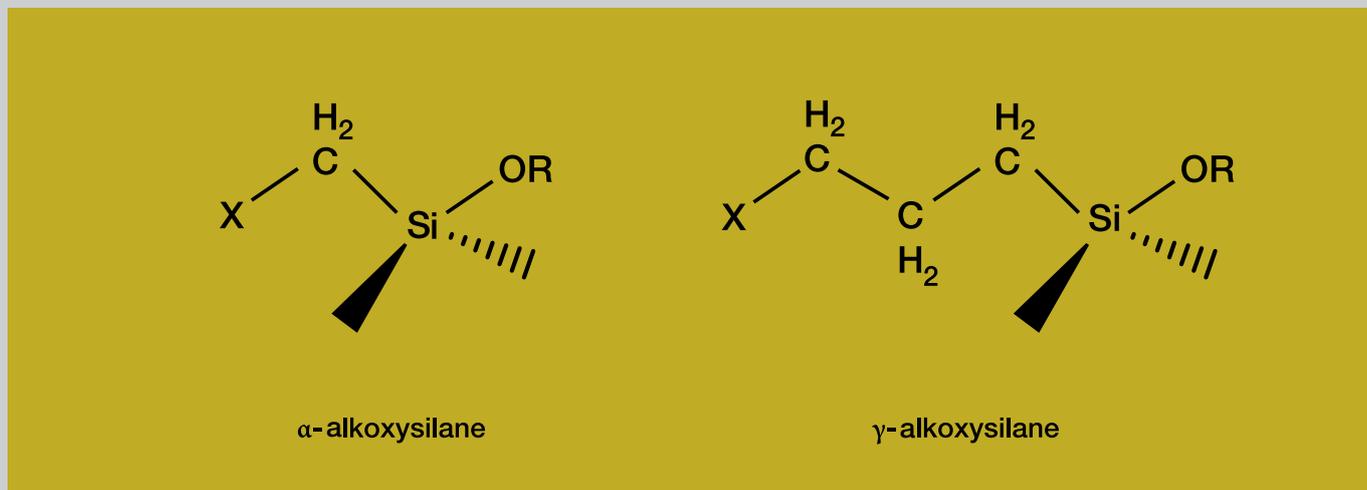
### High reactivity of the $\alpha$ -silanes

The relatively slow crosslinking kinetics of the standard silane-terminated polymers are due to the molecular structure of the terminal silyl group. With  $\gamma$ -alkoxysilanes, the moisture-induced crosslinking reaction takes place considerably more slowly than with the highly reactive  $\alpha$ -alkoxysilanes, which

have only recently become available on a commercial scale.

In  $\alpha$ -silanes, the electron donor is attached to the silicon atom via a methylene group. With this configuration, the alkoxy groups are activated, so that the crosslinking reaction is accelerated considerably ( $\alpha$ -effect). This is the decisive difference between  $\alpha$  and  $\gamma$ -silanes (**Box 1 and Box 2**).

Box 1:  $\alpha$ -alkoxysilane and  $\gamma$ -alkoxysilane



#### $\alpha$ - and $\gamma$ -alkoxysilanes

In organofunctional alkoxy-silanes, at least one of the four groups bound to the silicon atom is an alkoxy group ( $-OR$ ). Depending on the number of alkoxy groups, a distinction is made between bifunctional (two  $-OR$  groups) and trifunctional silanes (three  $-OR$  groups). The alkoxy groups are able to hydrolyse. The reaction with water leads to the formation of a siloxane network.

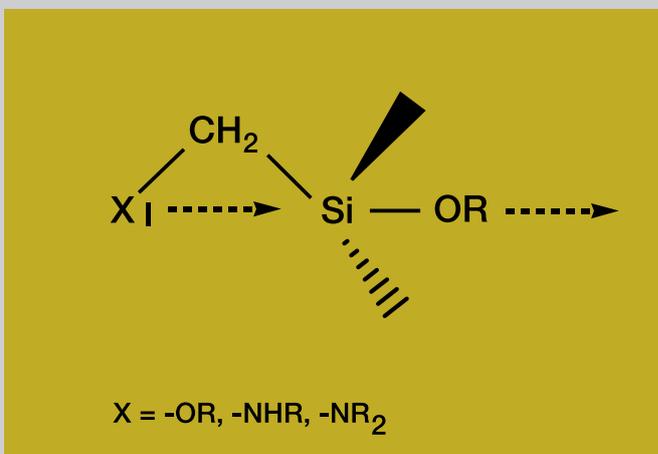
In addition to the alkoxy groups, there is a functional organic group on the silicon atom (a hydrocarbon chain with a reactive terminal X). Via X, a silane can also bind to an organic molecule. This is how silane-terminated polymers are produced.

An important structural feature of organofunctional alkoxy-silanes is the length of the hydrocarbon chain in the reactive organic group. The  $\gamma$ -silanes used industrially so far contain a propylene group in their reactive organic group ( $-CH_2-CH_2-CH_2-$ ) as spacer between the silicon atom and the organofunctional group, so that X is in the  $\gamma$ -position (relative to the Si atom). In  $\alpha$ -silanes, the considerably shorter methylene group ( $-CH_2-$ ) is located between the silicon atom and X. In this case, X is  $\alpha$ -terminal. The length of the hydrocarbon chain has a major influence on how firmly the alkoxy groups are bound to the silicon atom, and thus on the speed of crosslinking in the presence of moisture ( $\alpha$ -effect).

#### The $\alpha$ -effect

In  $\alpha$ -alkoxysilanes, the alkoxy groups are activated, so that these silanes are extremely reactive toward water. The reason for this effect is an electronic interaction between the silicon atom and the electron donor in the  $\alpha$ -position (usually a nitrogen atom with a free electron pair). As a result of back-bonding, the electron density is shifted from the donor to the silicon atom, and the Si – O bonds are weakened. The alkoxy groups split off more easily and hydrolysis is accelerated.

Box 2: Diagram showing the  $\alpha$ -effect in alkoxy-silanes



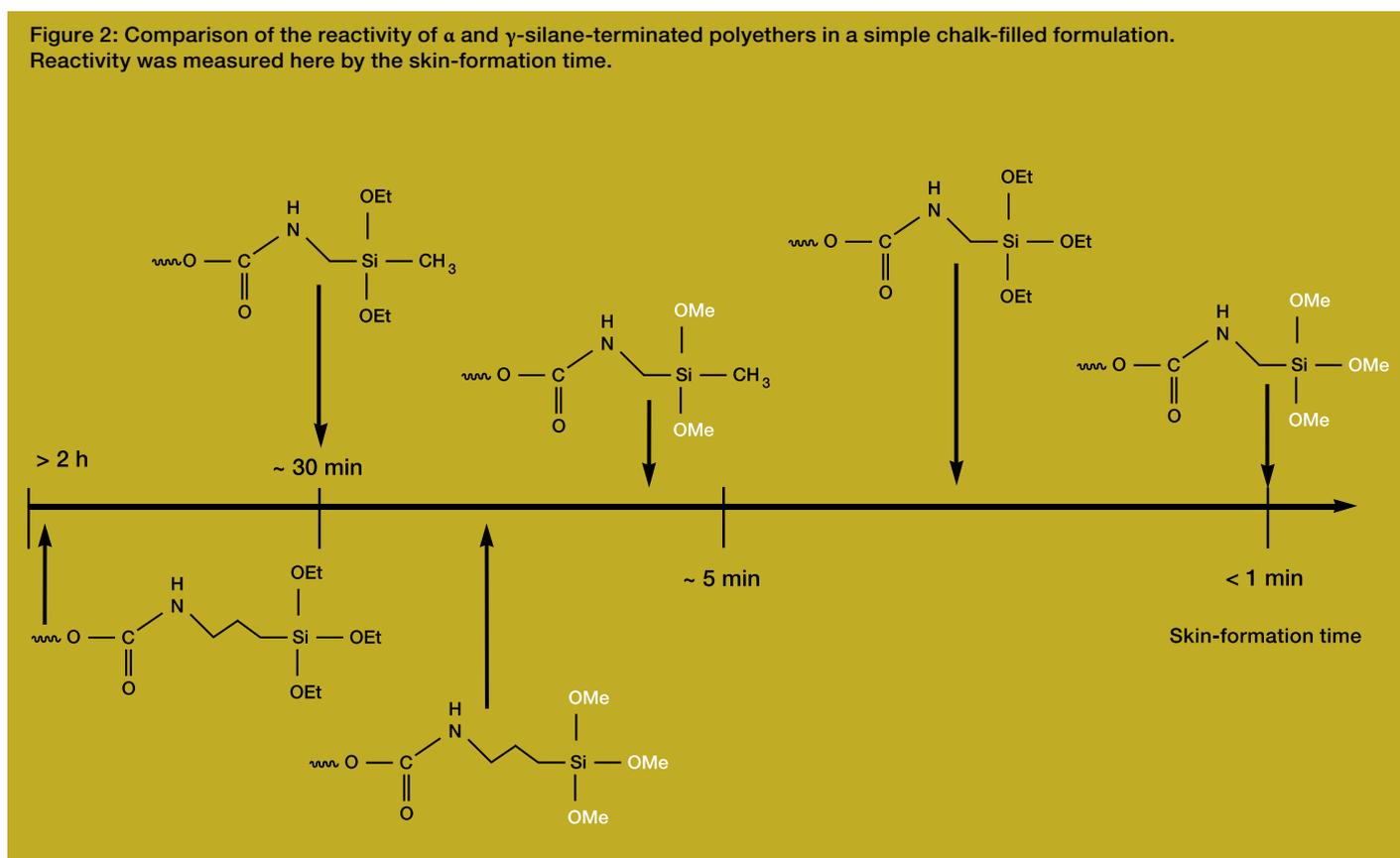
The high reactivity characteristic of  $\alpha$ -silanes is retained even when they are attached to organic polymers.  $\alpha$ -silane-terminated polymers always crosslink very quickly.

**Figure 2** illustrates this by way of skin-formation times, which were measured for various silane-terminated polyethers. Even dimethoxysilyl and triethoxysilyl end groups are more reactive in  $\alpha$ -silane-terminated polyethers than the trimethoxysilyl end groups of  $\gamma$ -silane-terminated polyethers. Difunctional silanes have the advantage that they release less methanol during the crosslinking process. In addition, a lower crosslinking density is obtained with them than with trifunctional silanes, thus favoring the formation of a more elastic vulcanizate.

Alkoxysilanes with an  $\alpha$ -terminal isocyanate group ( $\alpha$ -NCO silanes) have proven particularly useful in reacting with organic polymers (reaction: **Figure 3**). There are three reasons for this: first, the isocyanate group ( $-N=C=O$ ) permits bonding to various organic base polymers. Second, the bonding reaction is easy to control, and third, the reaction is quantitative. In the resulting  $\alpha$ -silane-terminated polymer, the terminal silyl groups are irreversibly joined to the polymer chain via a stable urethane bond; the product contains no free isocyanate groups.



**Figure 2: Comparison of the reactivity of  $\alpha$  and  $\gamma$ -silane-terminated polyethers in a simple chalk-filled formulation. Reactivity was measured here by the skin-formation time.**

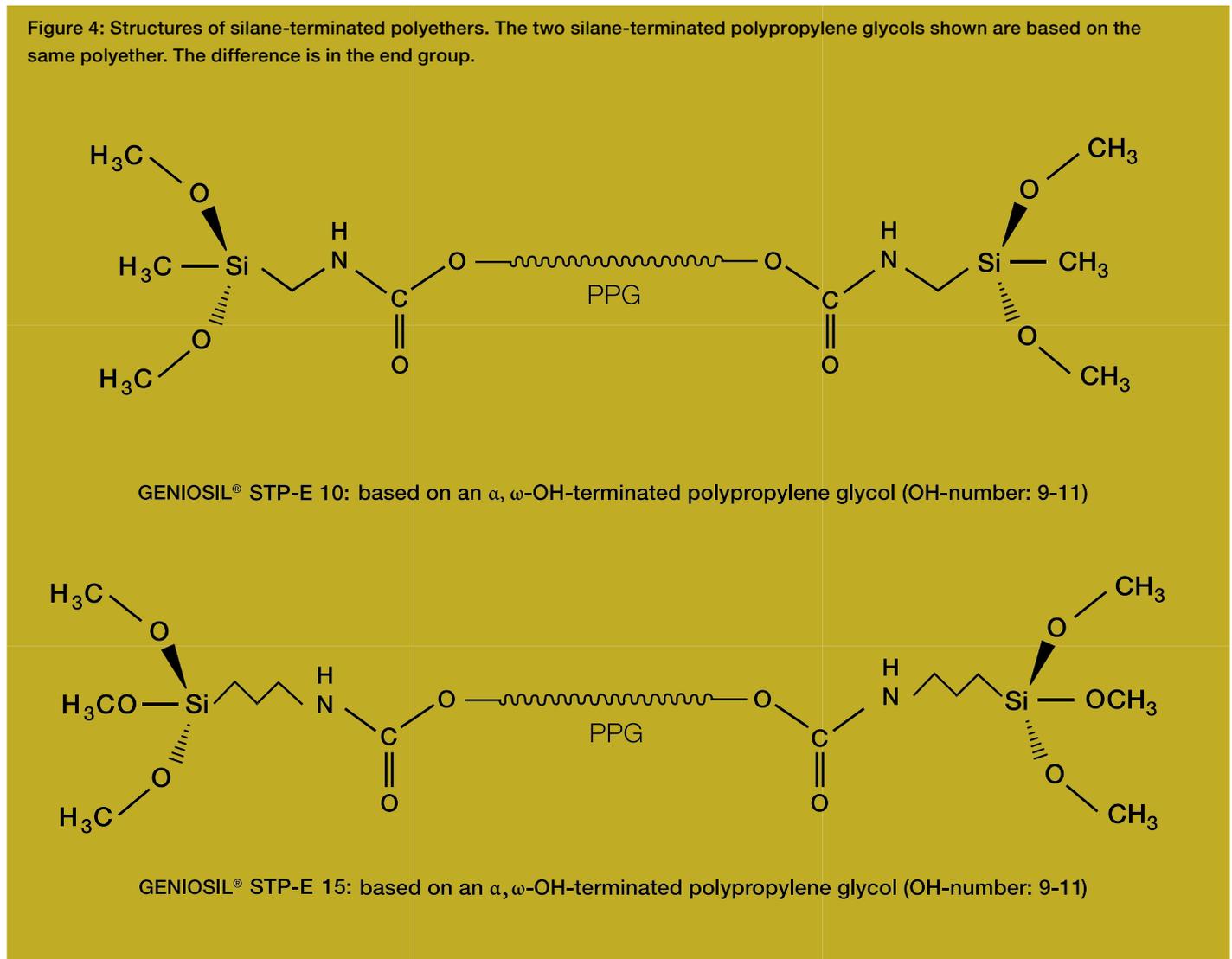




### Application-relevant properties

The molecules shown at the top in **Figure 2**, which are based on the polyether polypropylene glycol (PPG), are interesting functionalized polymers. Especially the polypropylene glycols terminated with an  $\alpha$ -dimethoxysilane – they appear in **Figure 2** with a skin formation time of about 5 minutes – have a property profile that makes them ideal for use as binders in elastic adhesives. These products are currently being launched on the market by WACKER SILICONES under the trade name GENIOSIL® STP-E (**Figure 4**).

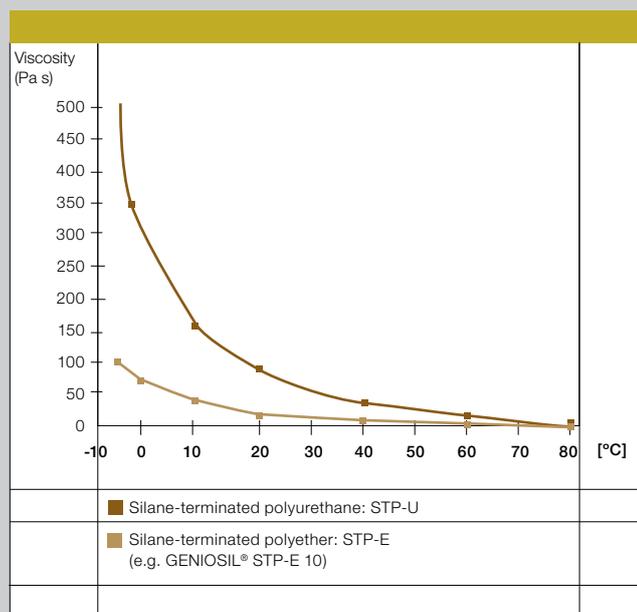
A manufacturing process was developed for the silane-terminated polymers based on isocyanatoalkoxysilanes using a special catalyst system that selectively catalyzes bonding of the silane to the polymer, but not hydrolysis of the alkoxy groups. This has a positive effect on the storage stability of the  $\alpha$ -silane-terminated polypropylene glycols and allows easy handling in the presence of atmospheric moisture.



\* GENIOSIL® is a trademark of Wacker Chemie AG

The main reason for using a polyether as the organic base polymer is that in polyethers – unlike polyurethanes – no hydrogen bonds form between the individual polymer chains. This explains their low viscosity, even after the silane termination. The viscosity of an  $\alpha$ -silane-terminated polyether is hardly any higher than that of a pure polyether of the same chain length, meaning that GENIOSIL® STP-E 10 (and comparably GENIOSIL® STP-E 15) has a viscosity of around 10 Pa s at room temperature (**Figure 5**).

**Figure 5: Viscosity of the silane-terminated polyether GENIOSIL® STP-E 10 and of a silane-terminated polyurethane with comparable properties in formulations.**



The absence of intermolecular hydrogen bonds also has a positive effect on the elasticity of the crosslinked  $\alpha$ -silane-terminated polyether. A crosslinked PPG does not become brittle even at very low temperatures.

The  $\alpha$ -silane-terminated polyethers are not known to be harmful in any way. They offer broad scope for formulating the adhesive and are easily adjusted to provide universal adhesion by adding standard organofunctional silanes as adhesion promoters; the use of a primer is seldom necessary.

Since  $\alpha$ -silane-terminated polyethers are highly reactive, catalyzing the crosslinking reaction is easy. The crosslinking kinetics can be controlled by a wide range of crosslinking catalysts. The reactivity and mechanical properties of the cured product (such as hardness and elasticity) can be influenced by adding  $\alpha$ -silanes as cross linkers or chain-extenders.

### Properties of formulations of silane-terminated polyethers

The high rate at which  $\alpha$ -silane-terminated polyethers crosslink is reflected in the skin-formation time. **Table 1** shows how the skin-formation time can be adjusted by varying the catalyst system. When a catalyst system containing tin is used (dibutyl tin dilaurate, DBTL), the  $\gamma$ -trimethoxysilane-terminated polyether (GENIOSIL® STP-E 15) crosslinks comparatively quickly. Because of its trifunctionality, its vulcanizate has a higher crosslinking density and thus lower elasticity than is achieved with difunctional end groups. Unlike this  $\gamma$ -product, an  $\alpha$ -silane-terminated polyether such as GENIOSIL® STP-E 10 will yield very fast-curing formulations without requiring an organotin catalyst. In the case of  $\alpha$ -silane-terminated polyethers, an aminosilane typically used as adhesion promoter (in the formulation shown this is N-Aminoethyl-aminopropyl-trimethoxysilane GENIOSIL® GF 91) will simultaneously assume the function of the crosslinking catalyst.

A formulation based on  $\alpha$ -silane-terminated polyethers cures very quickly, especially at the beginning of the crosslinking process. Even one-part formulations soon develop adequate strength – exactly how soon will depend on the size of the adhesive-air contact area (**Figure 6**) and other components in the formulation. The use of  $\alpha$ -silane-terminated polyethers thus has a positive influence on such important properties as green strength.

### Properties of the vulcanizate

Silane-terminated adhesives and sealants in which the base polymer has been modified with  $\alpha$ -silyl end groups cure rapidly and completely. Their vulcanizates are notable for high elasticity combined with high strength and hardness. Typical Shore A hardness figures are between 40 and 70; the hardness can be adjusted over a broad range by means of an appropriate formulation.

A fully cured  $\alpha$ -system has almost twice the elongation at break as an analogous  $\gamma$ -system, and even the tensile strength is somewhat better in the  $\alpha$ -system (see **Table 1**). Using adhesive formulations based on GENIOSIL® STP-E 10, tensile strengths at break of up to 5 N/mm<sup>2</sup> can be achieved, with considerably higher values possible in special formulations. **Table 2** provides an example showing how the mechanical properties of the vulcanizate change when different types of calcium carbonate fillers are used in the formulation.

The new  $\alpha$ -silane-terminated binders are particularly useful when high strength is required together with high elasticity. The elongation at break can be adjusted over a broad range, even well over 500 % and the Shore A hardness down to 20, by choosing the right fillers and plasticizers and using an appropriate quantity of suitable silane additives (chain extenders, water scavengers, catalysts).

**Table 1: Catalysis of silane-terminated polyethers. The reactivity and mechanical properties of polymers with  $\alpha$ -dimethoxy and  $\gamma$ -trimethoxy end groups are compared.**

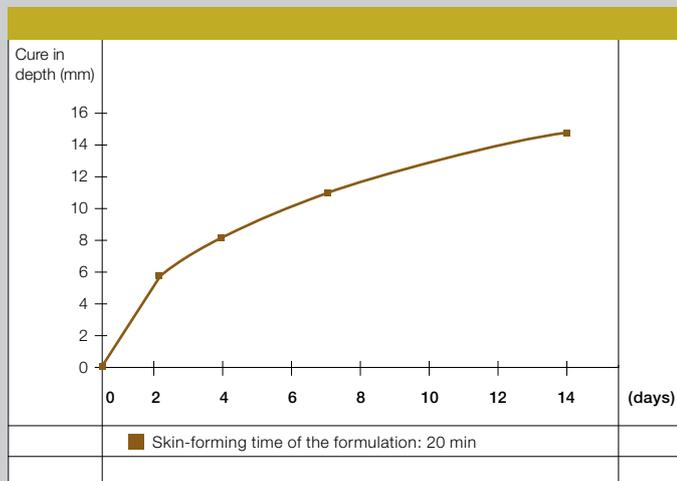
Formulation	GENIOSIL® STP-E 10 $\alpha$ -dimethoxy	GENIOSIL® STP-E 15 $\gamma$ -trimethoxy
<b>Reactivity - catalysis</b>		
1.0 % GF 91 <sup>1</sup> ; 0.25 % DBTL <sup>2</sup>	15 min	10 min
1.0 % GF 91 <sup>1</sup> ; 0.05 % DBTL <sup>2</sup>	30 min	> 3 h
2.0 % GF 91 <sup>1</sup>	50 min	> 6 h
1.0 % GF 91	70 min	> 6 h
Vulcanizate	tack free	tack free
<b>Mechanical properties of the cured polymer (DIN 53504)</b>		
Catalyst	1.0 % GF 96 <sup>3</sup>	1.0 % GF 96 <sup>3</sup> + 0.25 % DBTL <sup>2</sup>
Tensile strength at break (N/mm <sup>2</sup> )	0.7	0.6
Elongation at break (%)	70...90	40...50
Shore A hardness (DIN 53505)	40	50

<sup>1</sup> GENIOSIL® GF 91: N-Aminoethyl-aminopropyltrimethoxysilane  
<sup>2</sup> DBTL: Dibutyltindilaurate  
<sup>3</sup> GENIOSIL® GF 96: N-Aminopropyltrimethyltrimethoxysilane

**Table 2: Typical properties of silane-terminated polyether (GENIOSIL® STP-E 10) in formulations containing different chalk types (PCC = precipitated calcium carbonate, GCC = ground calcium carbonate - different basis).**

Formulation	1	2	3
Polymer - GENIOSIL® STP-E 10	38.5 %	39.5 %	39.5 %
Plasticizer - Diisodecylphthalate	10.0 %	10.0 %	10.0 %
Water scavenger (GENIOSIL® XL 63) <sup>1</sup>	3.0 %	1.0 %	1.0 %
Fumed Silica	9.0 %	9.0 %	9.0 %
PCC	38.5 %		
Coated GCC		39.5 %	
Coated GCC			39.5 %
Catalyst - dibutyltindilaurate	0.25 %	0.25 %	0.25 %
Catalyst/crosslinker (GENIOSIL® GF 91) <sup>2</sup>	0.75 %	0.75 %	0.75 %
Cured for two weeks at 23 °C/50 % rh			
100%-Modulus - N/mm <sup>2</sup> (DIN 53504-S1)	4.27	1.26	1.38
Shore A hardness (DIN 53505)	70	55	55
Elongation at break - % (DIN 53504-S1)	128	199	236
Tensile strength at break - N/mm <sup>2</sup> (DIN 53504)	4.8	1.8	2.4

<sup>1</sup> GENIOSIL® XL 63: N-Trimethoxysilylmethyl-O-methyl-carbamate  
<sup>2</sup> GENIOSIL® GF 91: N-Aminoethyl-aminopropyltrimethoxysilane



**Figure 6: Curing in depth as a function of time for a typical one-part formulation with GENIOSIL® STP-E 10.**

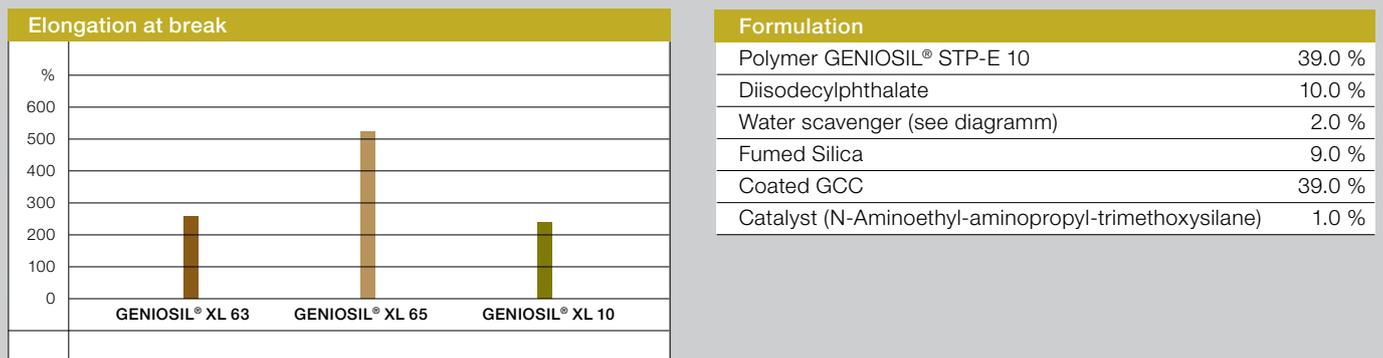
### Difunctional $\alpha$ -silanes as water scavenger and chain extenders

$\alpha$ -silanes can be used in sealant and adhesive formulations not only as crosslinkers but also as versatile compounding additives. Moisture in a formulation e.g. from fillers or by diffusion into the formulations upon storage of cartridges may lead to undesirable gelling. Typically STP formulations are protected against pre-curing by the addition of Vinyl-trimethoxy-silane (GENIOSIL® XL 10) or other reactive organofunctional silanes.  $\alpha$ -silanes however, such as N-Tri-methoxysilylmethyl-O-methyl-carbamate (GENIOSIL® XL 63) can speed up the reaction with moisture in the formulation without affecting the mechanical properties too much. By substitution of the trifunctional silanes and addition of the difunctional  $\alpha$ -silane N-Dimethoxy-methylsilylmethyl-O-methyl-carbamate (GENIOSIL® XL 65) elongation at break and the modulus of the system can be influenced without losing storage stability. The switch e.g. doubles the elongation at break

using the same amount of silane in the formulation as shown in **Figure 7**. This effect can be explained by a chain-elongation process of the difunctional silane which leads to higher molecular weight build-up or a lower crosslink density in the vulcanizate. This is only possible with  $\alpha$ -difunctional silanes.

Standard  $\gamma$ -difunctional silanes are usually too slow to interfere in the crosslinking reaction. Similar effects can be obtained by using  $\alpha$ -methacrylic silanes. In **Table 3** the strong effect on the modulus is shown by the addition of the two silanes Methacryloxymethyl-dimethoxy(methyl)silane (GENIOSIL® XL 32) and Methacryloxymethyl-diethoxy(methyl)silane (GENIOSIL® XL 34). Adding up to 2 % of silane to the formulations the modulus and Shore A hardness is lowered. In addition the elongation at break goes up. Larger amounts of difunctional methacrylic silanes already show a drop in tensile strength and curing of the end product is influenced (e.g. remains tacky over a longer period).

**Figure 7: Dimethoxymethylsilylmethyl-O-methyl-carbamate (GENIOSIL® XL 65) as chain extender for higher elasticity of the formulation.**



**Table 3: Formulations of STP-E using  $\alpha$ -methacrylic silanes as chain extender.**

Formulation	1	2	3	4
Polymer (STP-E)	39.0 %	39.0 %	39.0 %	39.5 %
Diisodecylphthalate	10.0 %	10.0 %	10.0 %	10.0 %
Water scavenger (GENIOSIL® XL 63)	1.0 %	1.0 %	1.0 %	1.0 %
Fumed Silica	9.0 %	9.0 %	9.0 %	9.0 %
Coated GCC	39.0 %	39.0 %	39.0 %	39.5 %
Crosslinker (GENIOSIL® XL 34)		2.0 %	1.0 %	
Crosslinker (GENIOSIL® XL 32)	2.0 %			
Catalyst (Dibutyltindilaurate)	0.1 %	0.1 %	0.1 %	
Catalyst/crosslinker (N-Aminoethyl-aminopropyl-trimethoxysilane)	0.5 %	0.5 %	0.5 %	0.75 %
Cured for two weeks at 23 °C/50 %rh				
100%-Modulus - N/mm <sup>2</sup> (DIN 53504-S1)	0.3	0.4	0.8	1.2
Shore A hardness (DIN 53505)	20	28	42	54
Elongation at break % (DIN 53504-S1)	529	604	485	284
Tensile strength at break N/mm <sup>2</sup> (DIN 53504-S1)	0.7	1.0	2.1	2.3

## Secondary $\alpha$ -Aminosilanes as crosslinker in silane-terminated polyurethanes

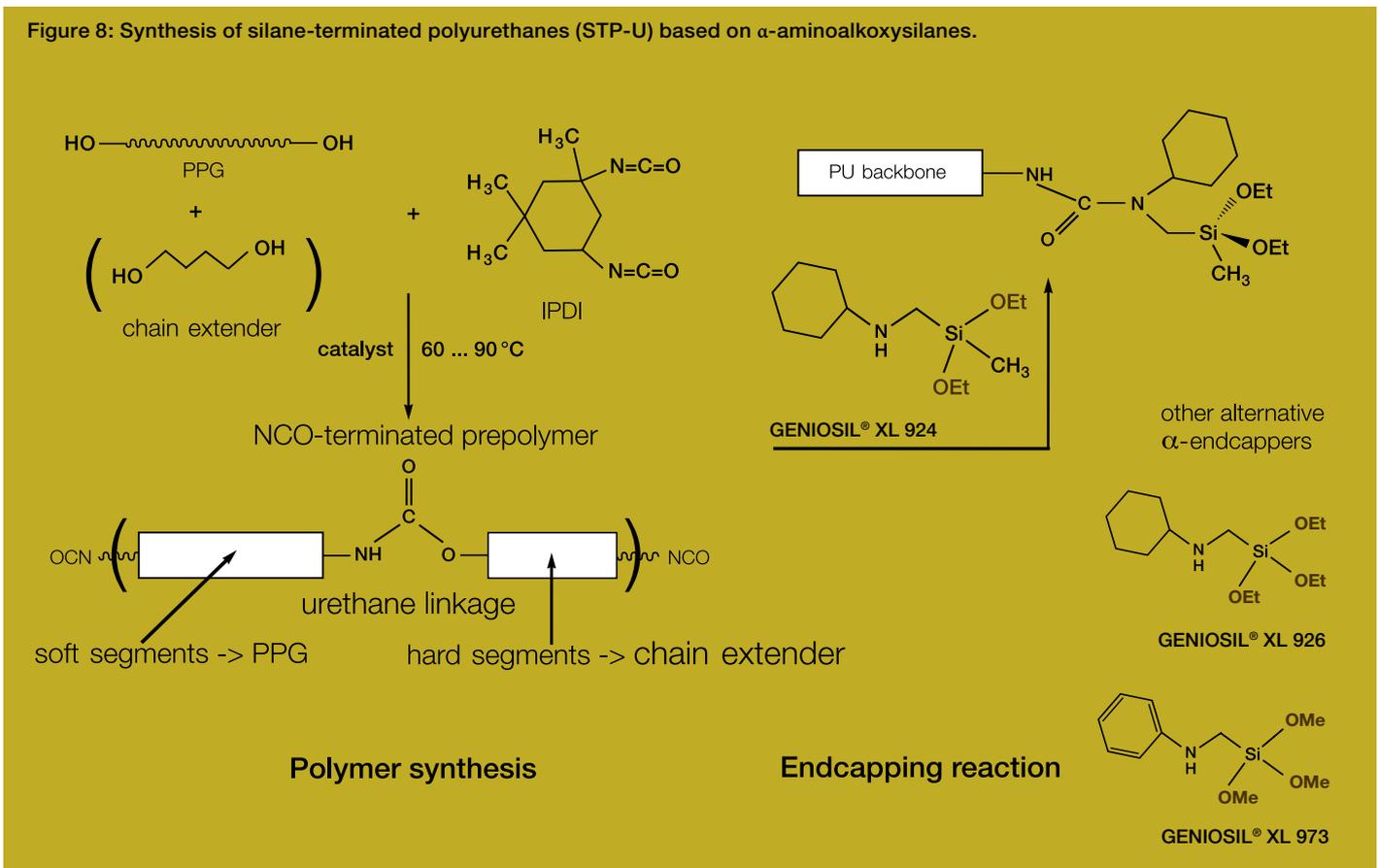
As mentioned earlier, silane terminated polymers are also feasible by the reaction of isocyanato-terminated polyurethane prepolymers with secondary aminosilanes. Secondary aminosilanes give – in contrast to primary aminosilanes – a clear and reproducible endcapping reaction. The properties of these polymers strongly depend on the nature of the prepolymer and the synthesis route. By using various polyurethane chemistry building blocks (e.g. diisocyanates, varying polyols, further chain extender) the mechanical properties of the silane-terminated system can be controlled. Nevertheless some limitations in the application are given by the viscosity of the endcapped polymer. In **Figure 8a** typical reaction is described.

In this example isophorone diisocyanate (IPDI) is reacted – typically using small amounts of a tin catalyst – with a linear  $\alpha$ - $\omega$  difunctional polypropylene glycol of higher molecular

weight (here approx. 8,000 and 12,000 - determined by the OH-value of the polymers). In addition a chain extender like 1,4-butanediol can be used to produce hard segments for higher mechanical strength. The NCO-terminated polymer is reacted in a second step with the aminosilane. Using primary silanes here leads to side reactions forming branched structures which normally result in higher viscosities or even gelling of the polymer. With secondary aminosilane the endcapping is a clean reaction with very few side reactions.

Using N-Cyclohexylaminomethyl-methyldiethoxysilane (GENIOSIL® XL 924) or N-Cyclohexylaminomethyl-triethoxysilane (GENIOSIL® XL 926) high reactive  $\alpha$ -silanes can easily be incorporated. With GENIOSIL® XL 924 and XL 926, polymers that cure rapidly but still only emit ethanol are feasible.  $\gamma$ -aminoethoxysilanes by contrast, are usually too slow for use as crosslinkers in adhesives. The properties of these  $\alpha$ -aminosilane terminated polyurethanes based on different building blocks and in a formulation are shown in **Table 4**.

Figure 8: Synthesis of silane-terminated polyurethanes (STP-U) based on  $\alpha$ -aminoalkoxysilanes.



**Table 4: Mechanical properties of alpha-silane based STP-U formulations.**

Polymer	1	2	3
Molecular weight - PPG <sup>(a)</sup>	12000	12000	8000
Isocyanate	IPDI <sup>(b)</sup>	IPDI	IPDI
Chain extender	diol <sup>(c)</sup>	diol	diol
NCO/OH ratio (prepolymer)	1.4	1.4	1.4
Silane endcapper	GENIOSIL® XL 924 <sup>1</sup>	GENIOSIL® XL 926 <sup>2</sup>	GENIOSIL® XL 924 <sup>1</sup>
NCO/NH ratio (endcapping)	1.6	1.6	1.6
Viscosity (Pa s/20 °C)	250	200	350
Cured for two weeks at 23 °C/50 % rh			
Polymer (%)	96	96	96
Crosslinker GENIOSIL® XL 63 <sup>3</sup> (%)	2	2	2
Catalyst GENIOSIL® GF 96 <sup>4</sup> (%)	2	2	2
Tensile strength at break N/mm <sup>2</sup> (DIN 53504-S1)	1.2	1.4	2.6
Elongation at break % (DIN 53504-S1)	520	400	570
100%-Modulus N/mm <sup>2</sup> (DIN 53504-S1)	0.3	0.4	0.6
Shore A hardness (DIN 53505)	25	30	50
<sup>1</sup> GENIOSIL® XL 924: N-Cyclohexylaminoethyl-methyldiethoxysilane		(a) Calculated from OH-value	
<sup>2</sup> GENIOSIL® XL 926: N-Cyclohexylaminoethyl-triethoxysilane		(b) IPDI = isophorone diisocyanate	
<sup>3</sup> GENIOSIL® XL 63: N-Trimethoxysilylmethyl-O-methylcarbamate		(c) diol = 1,4 butanediol	
<sup>4</sup> GENIOSIL® GF 96: Aminopropyl-trimethoxysilane			

## Conclusion and outlook

When an  $\alpha$ -silane-terminated polyether is used as binder, the fully cured adhesive offers a combination of strength and elasticity that is ideal for many demanding applications. The  $\alpha$ -effect makes the innovative binders highly reactive. The curing speed can be adjusted by the choice of catalyst system, and even tin-free catalyst systems are possible. The new binders give compounders great flexibility in formulating adhesives, not least because of the very low viscosities. They make it possible to produce formulations which possess the curing kinetics of reactive  $\alpha$ -silanes, while also closing the gap between typical silicone sealants and polyurethane systems in terms of their mechanical properties.

So much for the latest state of the art. The  $\alpha$ -silane technology has great potential, however, which is far from being exhausted. The  $\alpha$ -effect is so strong, for example, that even difunctional and trifunctional  $\alpha$ -ethoxysilanes are hydrolyzed sufficiently quickly. Attaching these silanes to organic polymers leads to ethanol-releasing binder systems as shown in formulations containing the silane-terminated polyurethane based on  $\alpha$ -aminoethoxysilanes.

Development work is also progressing in a second direction. The  $\alpha$ -isocyanatosilanes used to produce the  $\alpha$ -silane-terminated polyethers (GENIOSIL® STP-E) are suitable not only for bonding to polyethers, but can also be linked to other organic base polymers. This might give rise to new binders with a different property profile.

Formulation	
<b>Polymer 3</b>	57.5 %
Water scavenger (GENIOSIL® XL 63)	3.0 %
Fumed Silica	7.5 %
Coated GCC	30.0 %
Catalyst/Crosslinker (GENIOSIL® GF 96)	2.0 %
Cured for two weeks at 23 °C/50 % rh	
Tensile strength at break - N/mm <sup>2</sup> (DIN 53504-S1)	6.7 %
100%-Modulus - N/mm <sup>2</sup> (DIN 53504-S1)	720 %
Elongation at break - % (DIN 53504-S1)	2.0 %
Shore A hardness (DIN 53505)	70



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