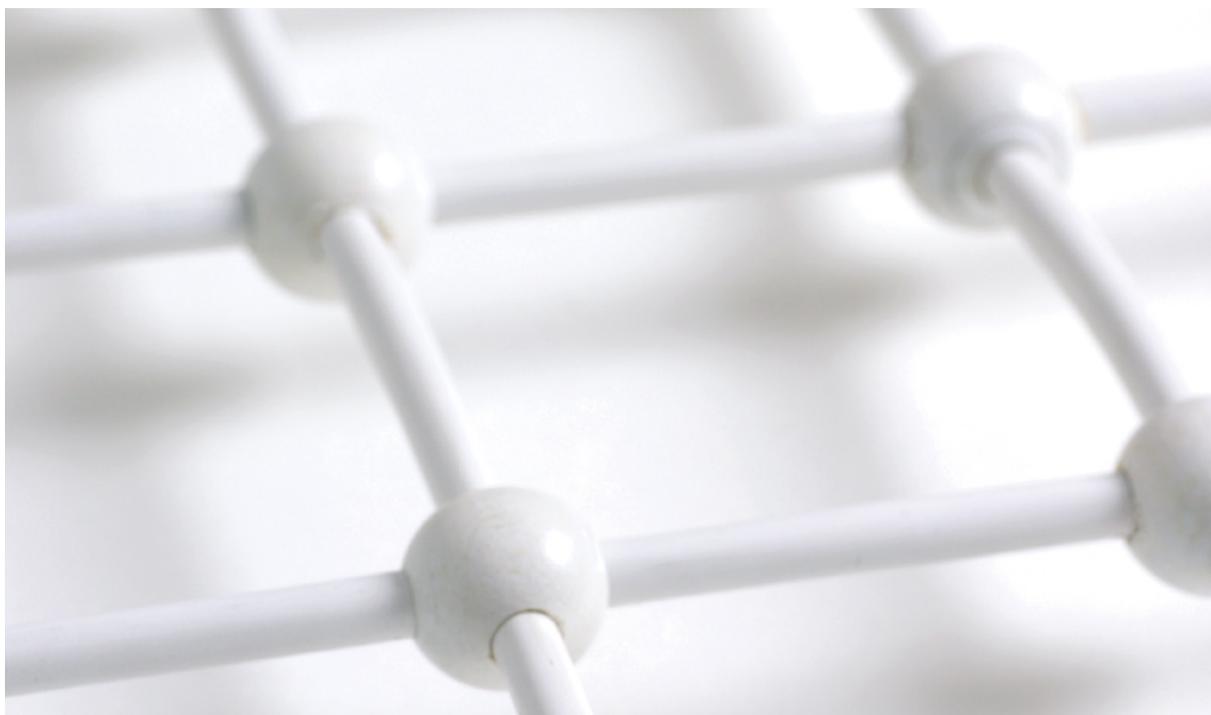
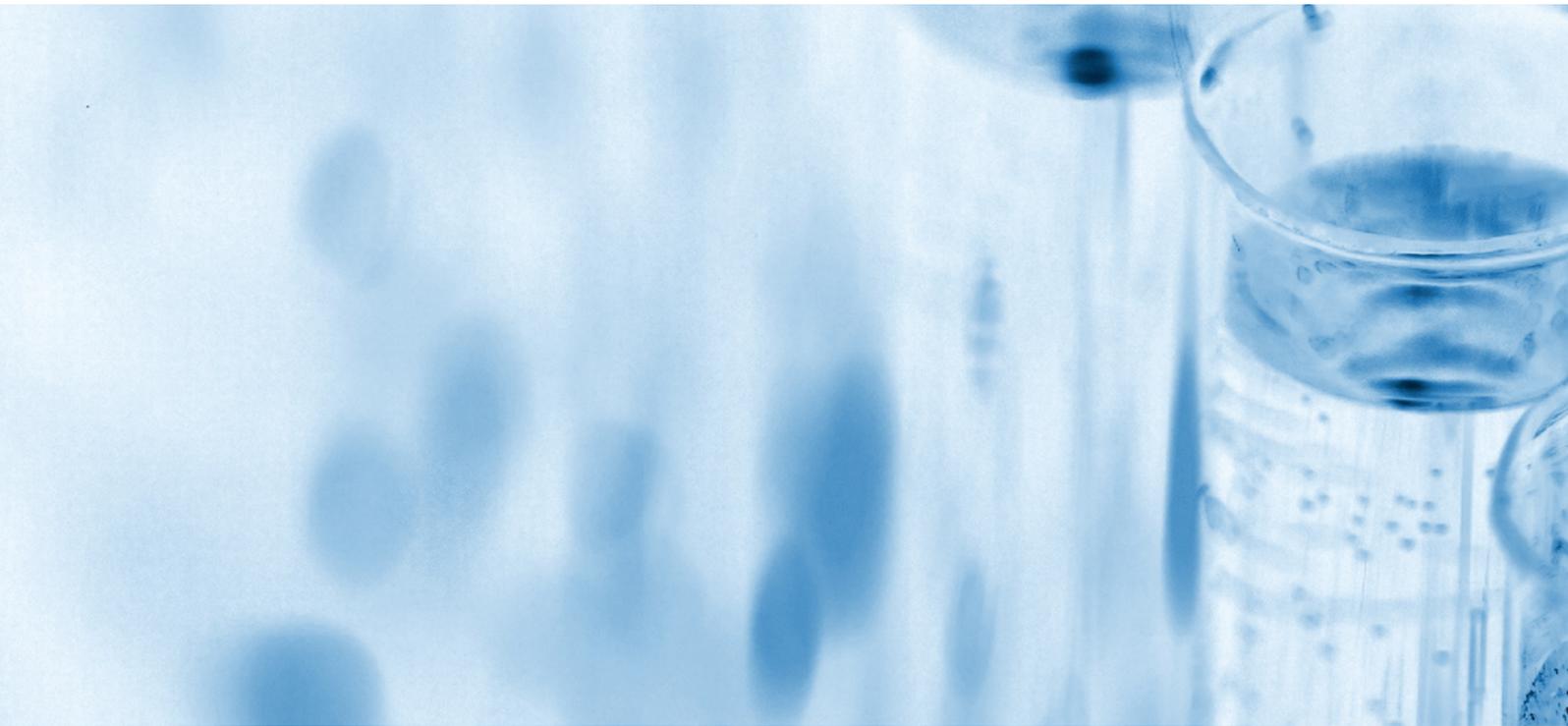


Dynasylan® Crosslinking of Polyethylene




Dynasylan®

 **EVONIK**
Leading Beyond Chemistry



Why Crosslink Polyethylene?

Polyethylene (PE), a thermoplastic polymer, is meltable and formable at elevated temperatures. For this reason, standard polyethylene grades are limited to applications with temperature maxima significantly below the melting temperature. A temporary temperature peak can lead to the complete failure of a product, for example, cable insulation.

Through crosslinking, the thermodynamic stability of polyethylene (PEX) can be significantly improved. This makes it possible to use PEX products in applications with higher temperature requirements that exceed the temperature stability of normal thermoplastic grades.



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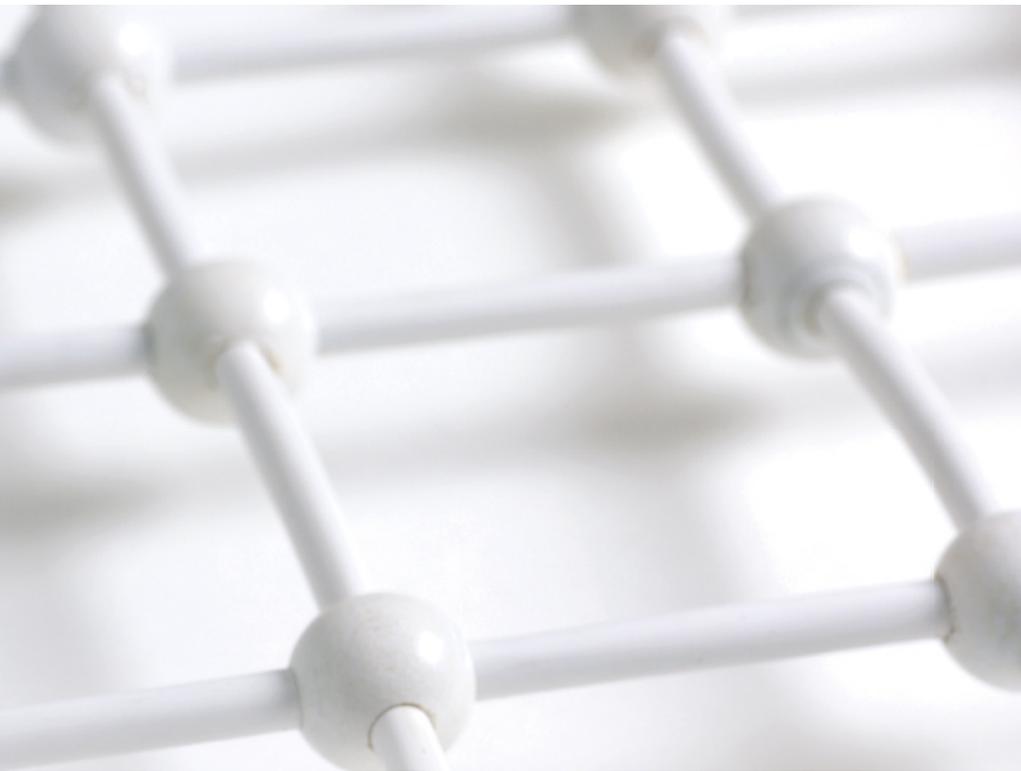
The use of Dynasylan® significantly improves the hot-set stability of polyethylene

At elevated temperatures, non-cross-linked polyethylene is easily deformed and thus displays poor mechanical properties. An important test is the hot set test at 200°C (392°F), where the deformation at elevated temperatures is observed.



Non-crosslinked polyethylene is very easily deformed as shown in the picture. Through silane crosslinking, the melted polymer retains its dimensional stability and the deformation of the crosslinked polyethylene is minimal (see right part of the picture). By contrast, non-crosslinked polyethylene breaks when exposed to such elevated temperatures (see left part of the picture).

Hot-set characteristic of polyethylene at 200 °C (392 °F), DIN EN 60811-2-1



Basic Methods for Crosslinking Polyethylene

Peroxide and radiation crosslinking



Polyethylene crosslinking through polyethylene radicals.

Both peroxide and electron beam crosslinking technologies for polyethylene are chemically very similar. Radicals are generated in both cases, which then recombine to form a chemical bond. This new chemical bond brings about the improvement in mechanical performance in the polyethylene that is associated with PEX.

Peroxide crosslinking technology requires the temperature profile to be within a tight operating window during processing.

It is especially important that the processing temperature does not exceed the decomposition temperature of the peroxide prematurely, otherwise the polymer will be crosslinked before the product has been shaped.

Electron beam crosslinking is generally associated with high processing costs, as it requires special equipment at high investment costs.

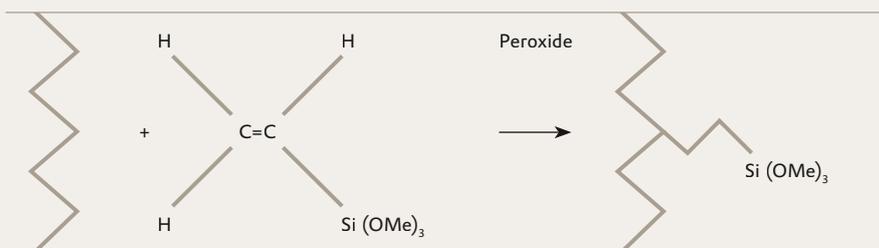
Silane crosslinking

In the last 30 years, silane crosslinking (PEX-b) has proven to be the most economical way to crosslink polyethylene for cables and pipes. Compared to other crosslinking technologies, such as peroxide (PEX-a) or radiation (PEX-c) curing, crosslinking by means of organofunctional silanes has the following major advantages:

- Well-established and economical process
- Environmentally friendly technology
- Crosslinking occurs outside the extruder
- Low investment costs
- High flexibility due to a broader processing window
- High throughput rates
- Small amounts of silane result in excellent properties

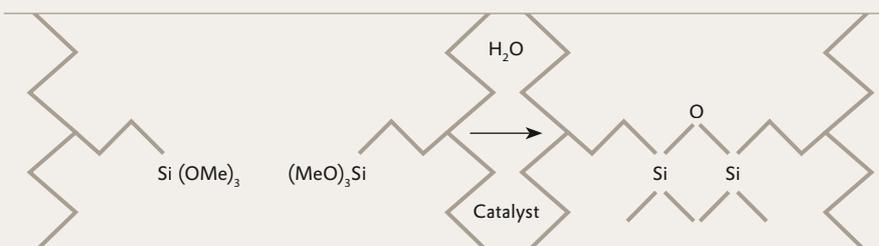
The crucial difference between silane technology and peroxide or electron beam processes is that the polymer is chemically modified in the first step. Generally, this reaction is carried out by grafting a vinylsilane onto the polymer chain with small amounts of peroxide. After grafting, the polymer is still thermoplastic and can be used in many ways. Crosslinking itself always takes place outside the extruder and is initiated by water, for example, in a water bath, steam chamber, or at ambient conditions. Silane technology has proven to be the most economical way to crosslink polyethylene.

Part A Grafting



Vinylsilane is attached to the polymer backbone through peroxide grafting.

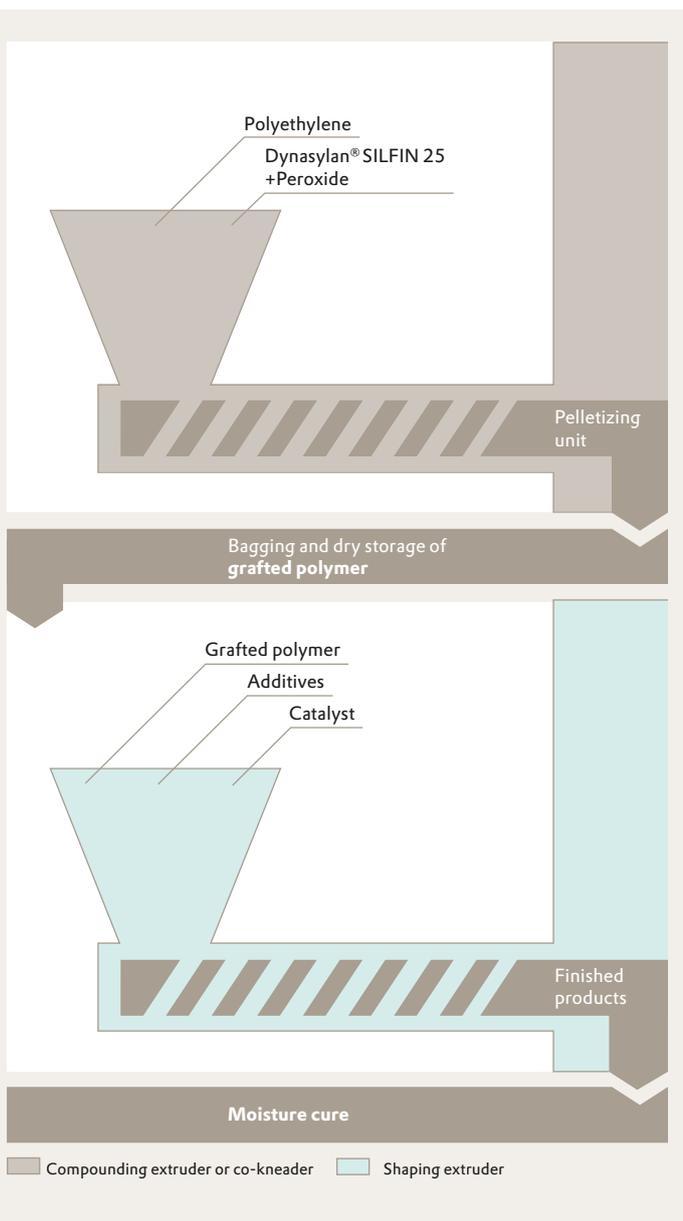
Part B Grafting



Crosslinking occurs through condensation of silane species.



Silane Crosslinking Technologies – Grafting and Shaping



Sioplas® process

The Sioplas® process was developed in 1968 by Midland Silicones. The essential step is peroxide-activated grafting of the vinylsilane onto the polymer chains. This step is usually carried out in compounding units such as co-kneaders or twin-screw extruders. The silane/peroxide mixture is added to the polymer melt by means of membrane or piston pumps, giving a grafted granular product.

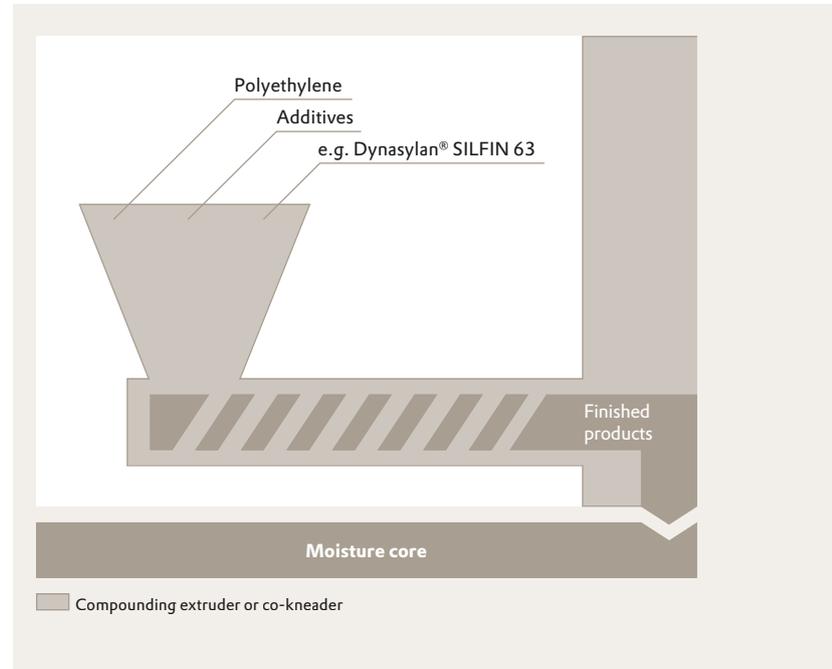
The grafted compound can be shaped on conventional extruders. The catalyst masterbatch, which increases crosslinking speed, needs to be added in this step.

Silane copolymers

Crosslinkable polymers can also be obtained by copolymerizing ethylene and vinyltrimethoxysilane. Such grades are supplied directly by polymer producers. The final shaping step is carried out, in a way similar to the Sioplas process, on standard extrusion equipment. The addition of a catalyst and further additives (e.g., stabilizers) in the shaping step is crucial.

Monosil® process

The Monosil® process developed by BICC and Maillefer in 1974 combines grafting, the addition of the catalyst, and shaping into one step. This significantly challenging process combination requires special extrusion equipment. Crucial aspects of the equipment are the screw design and the technology for feeding the additives.



Silane Crosslinking Technologies – Moisture Cure

Water is essential for the final crosslinking step. The water molecules have to penetrate the polymer and must initiate the hydrolysis reaction, which is followed by condensation of the resulting silanol species. The highest possible amount of water molecules on the polymer surface, in combination with high temperatures, is the optimal solution for the penetrating water and the crosslinking reaction. As a logical outcome, the most effective and fastest way is the use of a waterbath at elevated temperatures (80–90°C / 176–194°F at ambient pressure).

Furthermore, the speed can be increased significantly by using higher pressure. However, this method requires extra investment. One other option is to use steam. But this kind of crosslinking is generally slower than a water bath. Crosslinking under ambient conditions is the most convenient method, but it generally requires significantly longer reaction times.

Crosslinking of Cables at Ambient Conditions

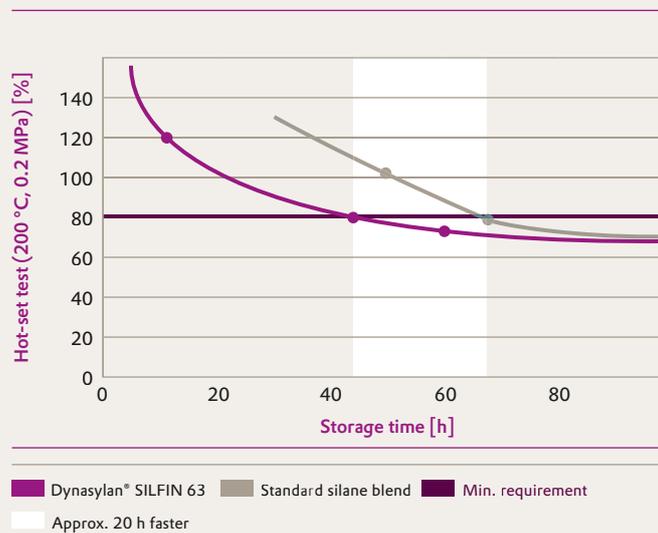
The crosslinking speed of silane modified polyethylene is affected by:

- Silane grade
- Number of water molecules present
- Temperature
- Pressure
- Grafting efficiency
- Amount of catalyst
- Dimensions of part to crosslink

For crosslinking at ambient conditions, the challenge is that the amount of water available is limited and the temperature is significantly reduced to standard crosslinking methods. Nevertheless, crosslinking must take place. Thus, the challenge is to find a system that increases the crosslinking speed at low temperatures and low humidity, but without pre-scorch during the extrusion step.

Compared to standard systems, Dynasytan® SILFIN 63 significantly increases the crosslinking speed of PEX cable insulation at ambient conditions (see diagram).

Improved performance of Dynasytan® SILFIN 63



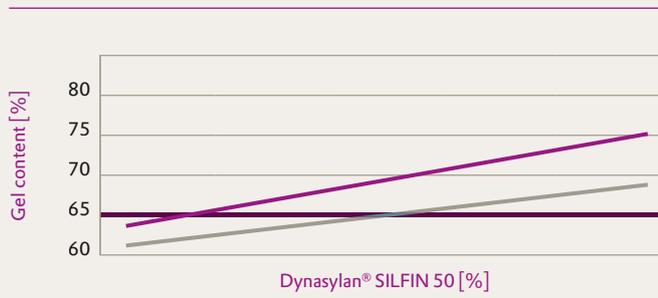
Dynasytan® SILFIN 63 achieves minimum requirement for hot-set test significantly faster.

Silane Crosslinking of Polyethylene-HD Pipes

Silane crosslinked polyethylene pipes show much better temperature creep behavior than non-crosslinked polyethylene-HD pipes, thereby allowing long-term service temperatures of 90°C. Other crucial advantages of crosslinked polyethylene for pipes are:

- Improved chemical and corrosion resistance; PEX does not dissolve in solvents
- Significant reduction in weight
- Significant reduction in laying costs
- High abrasion resistance
- High thermal resistance
- High dimensional resistance
- Enhanced safety against crack formation
- Superior resistance against rapid crack propagation (RCP)
- Use in "no-dig" applications
- No corrosion like in metals

Polyethylene-HD pipes can be efficiently crosslinked with Dynasylan® SILFIN 50



■ Polymer A ■ Polymer B ■ Min. requirement

Depending on the type of HD-Polyethylene, the amount of Dynasylan® SILFIN 50 needs to be varied in order to achieve the 65 % gel-content level.

For the manufacture of the polyethylene pipes used for potable water supplies, Dynasylan® VTMO is an ideal crosslinker. In many countries, Dynasylan® VTMO has received a document of compliance issued

by the regulatory agencies. If you have any specific questions, please use our solution finder on www.dynasylan.com or contact your local sales representative.

Product Overview

Product name	Description and use	Applications			Crosslinking		Coupling							
		Cables	Pipes	Filled plastics compounds	1-step (Monosil®)	2-step (Sioplas®)	Polyethylene	EVA/Polyethylene	Polypropylene	Rubber	Acrylics	Epoxy	Unsaturated polyester	Others
Multifunctional Silane Systems™														
Dynasylan® SILFIN 06	Standard product for cables	•			•									
Dynasylan® SILFIN 13	Standard product for cables	•				•								
Dynasylan® SILFIN 22	Standard product for cables	•			•									
Dynasylan® SILFIN 25	Multifunctional Silane System™, enables higher throughput	•	•		•	•								
Dynasylan® SILFIN 50	Multifunctional Silane System™ for pipes		•		•									
Dynasylan® SILFIN 53	Multifunctional Silane System™ for cables, LLDPE	•			•									
Dynasylan® SILFIN 63	Multifunctional Silane System™ for cables, ambient curing	•			•									
Dynasylan® SILFIN 70	High-performance Multifunctional Silane System™ for crosslinking of HFFR cables			•	•									
Dynasylan® SILFIN 71	High-performance Multifunctional Silane System™ for crosslinking of HFFR cables			•	•									
Dynasylan® SILFIN 75	High-performance Multifunctional Silane System™ for crosslinking of cables	•			•									
Dynasylan® SILFIN 80	All-in-one Multifunctional Silane System™ for cables	•			•									
Dynasylan® SILFIN 100	DBTDL-free alternative to SILFIN 06	•			•									
VPS SILFIN 201	Tin-free Multifunctional Silane System™ for crosslinking of cables	•			•									
VPS SILFIN 202	Tin-free Multifunctional Silane System™ for crosslinking of cables	•			•									
Aminosilanes														
Dynasylan® AMEO	Coupling agent for polar compounds			•				•	•					
Dynasylan® SIVO 214	High-performance Multifunctional Silane System™ for polar compounds			•				•	•					
Dynasylan® SIVO 210	High-performance Multifunctional Silane System™ for polar compounds			•				•	•					
Dynasylan® 1189	High-performance silane for polar compounds			•				•	•					
Dynasylan® HYDROSIL 1151	Waterborne, VOC-free high-performance Multifunctional Silane System™ for polar compounds			•				•	•					
Dynasylan® HYDROSIL 2775	Waterborne, VOC-free high-performance Multifunctional Silane System™ for polar compounds			•				•	•					

Product Overview

Product name	Description and use	Applications			Crosslinking		Coupling							
		Cables	Pipes	Filled plastics compounds	1-step (Monosil®)	2-step (Sioplas®)	Polyethylene	EVA/Polyethylene	Polypropylene	Rubber	Acrylics	Epoxy	Unsaturated polyester	Others
Vinylsilanes														
Dynasylan® VTMO	Vinylsilane	•	•	•	•	•								
Dynasylan® VTEO	Vinylsilane		•	•	•	•								
Dynasylan® VTMOEO	Vinylsilane			•			•	•		•				
Dynasylan® 6490	High-performance Multifunctional Silane System™ for non-polar compounds			•			•	•		•				
Dynasylan® 6498	High-performance Multifunctional Silane System™ for non-polar compounds			•			•	•		•				
Dynasylan® 6598	High-performance Multifunctional Silane System™ for non-polar compounds			•			•	•		•				
Dynasylan® HYDROSIL 2907	Waterborne, VOC-free high-performance Multifunctional Silane System™ for polar compounds			•			•	•		•				
Alkylsilanes														
Dynasylan® 9896	Hydrophobation agent			•										•
Dynasylan® OCTEO	Hydrophobation agent			•										•
Dynasylan® IBTEO	Hydrophobation agent			•										•
Dynasylan® HYDROSIL 2776	Waterborne, VOC-free hydrophobation agent			•										•
Dynasylan® HYDROSIL 2909	Waterborne, VOC-free hydrophobation agent			•										•
Phenylsilanes														
Dynasylan® 9165	High-performance silane for high temperature polymers			•										•
Dynasylan® 9265	High-performance silane for high temperature polymers			•										•
Fluorosilanes														
Dynasylan® F8261	High-performance silane for fluorinated polymers			•				•	•					•
Dynasylan® HYDROSIL F8815	Waterborne, VOC-free hydrophobation agent			•										•
Other functional silanes														
Dynasylan® MEMO	Coupling agent for unsaturated compounds									•	•	•	•	•
Dynasylan® GLYMO	Coupling agent for polar compounds									•	•	•	•	
Dynasylan® 4144	High-performance silane for hydrophilic applications									•	•		•	•
Dynasylan® 4148	High-performance silane for hydrophilic applications									•	•		•	•
Dynasylan® HYDROSIL 2926	Waterborne, VOC-free high-performance Multifunctional Silane System™ for hydrophobation and oleophobation			•				•	•					•

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