

# **Neuburg Siliceous Earth**

# in heavy-cuty 2K PU coatings,

# e.g. for pipelines

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#### 1 Introduction

2K-PU coatings are used as thick-layer up to millimeter scale, hard coatings for buried pipelines, predominantly for complex shaped parts such as bends or fittings and also as repair coatings and for transitions of pipes.

Compared with 2K epoxy systems, a comparable area can be coated with a higher layer thickness in a shorter time. The drying process and thus the usability or recoatability is much shorter (a few minutes to hours as opposed to several days). Possible defects can be detected earlier due to the rapid curing, and the temperature window for application is also larger.

Thus, if one considers not only the pure material costs, but also takes into account the application time, maintenance costs and other hidden costs, the use of 100 % PU systems is ultimately significantly more cost-effective.

The application is usually carried out in a 2K airless process. In addition to good deaeration and storage stability, important properties include good corrosion protection, a fast reaction (pot life < 5 min) and high requirements for mechanical properties such as strength, toughness and flexibility.

Sillitin Z 86 puriss is traditionally used in such systems.

The study shows the possibilities of using Neuburg Siliceous Earth as a mineral filler in a hard 2K polyurethane coating and looks at the differentiation of the individual siliceous earth grades.

Besides rheology and storage stability of the A-component, the mechanical properties of the cured formulations were also considered.

#### 2 Experimental

#### 2.1 Base formulation and filler combinations

The table gives a description of the structure of the base formulation. The A-component contains two different polyol types as binders, zeolite paste as molecular sieve for moisture absorption, some additives for defoaming/deaerating as well as wetting/dispersing agents and the catalyst. Also included is a combination of several fillers.

The B-component (hardener) is an isocyanate based on MDI prepolymer.

The mixing ratio of the two components is 1:1 by volume. This corresponds to an isocyanate excess of 25 %.

	Base Formu	lation	<b>HÖFFMANN</b> MIINIERAL
	Component A		parts by weight
	Desmophen BT 1400	Binder, polyol	13.14
	Desmophen T 460	Binder, polyol	13.14
EXPERIMENTAL	Combination of fillers		21.90
RESULTS	Finma Sorb 430 PR	Molecular sieve	5.21
	Ethacure 100 Plus	Crosslinking aid	1.26
SUMMARY	Byk-A 530	Defoamer	0.26
APPENDIX	Disperbyk 163	Wetting/dispersing agent	0.26
	Dabco LV 33	Catalyst	0.24
	Total Component A		55.41
	Component B		
	Desmodur E 29	Hardener, isocyanate (prepolymer based on MDI)	approx. 44
	Total Formulation A	+ B	approx. 99.4
MAR R	Mixing ratio		
	Component A:B by vo	1:1	
	Stoichiometric, isocya	nate/polyol	approx. 1.25
	VM-0/0321/03.2021		

The filler combination in the base formulation consists of barite, a natural mica-chlorite-quartz intergrowth (leucophyllite) and a quartz flour surface treated with epoxy silane. In the tests, the leucophyllite and the quartz flour were replaced by products of Neuburg Siliceous Earth in equal weights. The barite remained present in all formulations and was dosed unchanged.

	Filler Combinati	H <b>OFFMANN</b> MINIERAL		
INTRODUCTION EXPERIMENTAL				Variation with
RESULTS SUMMARY		Control [pbw]		Siliceous Earth [pbw]
APPENDIX	Barite	8.87		8.87
	Coalescence of mica, chlorite and quartz	6.69	replaced by weight with	12.02
	Quartz flour surface treated	6.34	Neuburg Siliceous Earth	13.03
Color St	VM-0/0321/03.2021			

# 2.2 Filler characteristics

The table summarizes the fillers used and shows their most important characteristic properties.

	Filler Characteristics							
INTRODUCTION		Particle size		Oil absorp- tion	Density	Specific surfacce BET	Surface treatment	
EXPERIMENTAL RESULTS		d <sub>50</sub> [µm]	d <sub>97</sub> [µm]	[g/100g]	[g/cm³]	[m²/g]		
	Coalescence of mica, chlorite and quartz	9	30	29	2.78	4.2		
	Quartz flour surface treated	4	11	24	2.65	3.0	Epoxy silane	
	Sillitin Z 86 puriss	1.9	9	55	2.6	12		
	Silfit Z 91	2.0	10	65	2.6	10		
	Aktifit PF 115	2.0	10	60	2.6	9	amino functionalized	
	Other fillers in the formulation (only for comparison)							
	Barite	2.9	14	14	4,4	1,7		
A States	VM-0/0321/03.2021							

Sillitin Z 86 puriss is a classic Neuburg Siliceous Earth grade and is already used by various customers as a cost-effective filler in such systems.

Other products used are Silfit Z 91 and Aktifit PF 115. Both are calcined grades, Silfit Z 91 being the untreated base grade. Aktifit PF 115 is surface treated with an amino functional group and has a hydrophobic surface character.

All Neuburg Siliceous Earth grades show clearly finer particles, which is consistently true for the mean particle diameter  $d_{50}$  as well as for the topcut  $d_{97}$  in the case of the mica-chloritequartz intergrowth. Also, Neuburg Siliceous Earth offers a significantly larger surface area as well as oil number and thus more interaction and anchoring potential for the polymer matrix. In contrast, the difference in density is negligible.

# 2.3 Preparation of A-component



### 2.4 Preparation of test specimens

Tensile specimens type 1A according to DIN EN ISO 527 were produced for testing tensile properties, as well as standard bars measuring 80 x 10 x 4 mm for assessing impact strength.

The stainless steel molds were pretreated with "Wax release agent GP" from Gößl & Pfaff. By means of a 2-component cartridge with a volume ratio of 1:1, the formulations were injected into the corresponding molds. The mixing of the two components took place in a helix mixing tube with a diameter of 8 mm and 32 mixing segments (see also the following figure). Immediately afterwards, the surface of the test specimens was smoothed with a metal blade. After curing for at least 3 hours or on the following day, the specimens could be demolded. The mechanical tests were performed after a total curing time of 14 to 16 days. Before testing, the edges of the specimens were deburred with fine sandpaper.



#### 3 Results

#### 3.1 **Properties A-component**

#### 3.1.1 Viscosity

7 days after preparation of the A-component, a flow curve from 0.1 to 1000 s<sup>-1</sup> was measured by means of logarithmically increased measuring points and the viscosity at 0.1 and 1000 s<sup>-1</sup> was evaluated.



The high shear rate (shaded columns) represents the behavior during pumping processes as well as the behavior during application. Here, hardly any difference between the formulations is detectable.

The low shear rate (filled columns) represents the behavior of the formulations after application, quasi at rest, and provides information on important properties such as stability and sagging tendency.

The formulation with Sillitin Z 86 puriss remains almost at the level of the control formulation, Silfit Z 91 tends to a somewhat stronger thickening effect. Aktifit PF 115 shows a marked increase in viscosity in the low shear range, corresponding to a higher stability or reduced sagging tendency.

In the graph, only the rheology of the A component is shown, since due to the very high reaction rate when the hardener is added, a measurement of the mixture is not accessible. If the low viscosity hardener (approx. 220 mPa·s) is added to the formulations in a volume ratio of 1:1, the viscosity level will be more than halved.

The formulation with Aktifit PF 115 nevertheless still shows a comparatively clearly higher low shear viscosity, which allows the application of higher film thicknesses without sagging tendency in one pass. This was found during the application into the mold.

### 3.1.2 Storage stability

The samples were stored at 40  $^\circ C$  for 3 months. The height of the supernatant and the solid sediment formed were assessed.

	Storage Stability A-Component						
	3 months at 4	O°C					
INTRODUCTION		Control	Sillitin Z 86 puriss	Silfit Z 91	Aktifit PF 115		
EXPERIMENTAL	Supernatant [%]	44	6	8	9		
RESULTS	Sediment [%]	5	3	5	none		
SUMMARY							
APPENDIX							
	VM-0/0321/03.2021						

Compared with the control formulation, the phase separation with Neuburg Siliceous Earth is clearly reduced. As with the control formulation, however, also with Sillitin Z 86 puriss and Silfit Z 91 a relatively solid sediment is formed over the storage time. With Aktifit PF 115, sediment formation can be completely prevented. The formulation is thus much easier to homogenize and thus perfectly storage stable.

# 3.2 Mechanical properties

### 3.2.1 Hardness

The Shore D hardness test was carried out according to DIN EN ISO 868 on the shoulders of the tensile specimens with a penetration time of the indenter of 15 s.



No difference can be detected between the formulations.

### 3.2.2 Tensile modulus

Representing the stiffness of the material, the tensile modulus was determined according to DIN EN ISO 527 at a test speed of 0.5 mm/min.



All formulations show about the same stiffness.

#### 3.2.3 Tensile strength and tensile strain at break

The test was carried out according to DIN EN ISO 527 with a test speed of 5 mm/min until the specimens break.



Also with regard to tensile strength, no difference can be observed between the formulations.



With Neuburg Siliceous Earth, a marked increase in tensile strain at break is obtained, especially with the two calcined grades. Silfit Z 91 and Aktifit PF 115 achieve an approx.

80 % increase in tensile strain at break and thus indicate a higher formability and flexibility of the coating.

#### 3.2.4 Impact strength

The test was performed according to DIN EN ISO 179-1 on unnotched specimens with a 4J pendulum.



While the formulations with the non-surface treated Neuburg Siliceous Earth grades Sillitin Z 86 puriss and Silfit Z 91 are on a somewhat lower or comparable level as the control formulation, the impact strength with Aktifit PF 115 increases markedly by approx. 30 %.



Neuburg Siliceous Earth is very well suited as a functional, mineral filler for heavy-duty 2K PU coatings.

The selected products can be easily incorporated and dispersed as mineral fillers.

Already the cost-effective Sillitin Z 86 puriss offers advantages in terms of storage stability and tensile strain at break compared to the filler combination of the mica-chlorite-quartz intergrowth (leucophyllite) and the surface-treated quartz flour used in the control formulation.

Silfit Z 91 and Aktifit PF 115, the two calcined Neuburg Siliceous Earth grades, give a further increase in tensile strain at break.

Aktifit PF 115 is also characterized, besides the improved impact strength, by the rheological activity. This allows the application of higher film thicknesses without sagging tendency and, as a positive side effect, prevents the formation of sediment during storage of the A-component.

In addition, the corrosion protection effect of the coating with Neuburg Siliceous Earth products should be improved, which would be expected for the hydrophobic Aktifit PF 115 in particular.

#### Product recommendations:

Sillitin Z 86 puriss	<ul> <li>+ cost-effective standard product</li> <li>+ improved storage stability</li> <li>(significantly less supernatant / slightly reduced sediment)</li> <li>+ improved tensile strain at break</li> </ul>
Silfit Z 91	+ color neutral
	+ improved storage stability (significantly less supernatant)
	+ significantly improved tensile strain at break
Aktifit PF 115	+ color neutral
	+ high rheological activity (non-sag behavior)
	+ strongly improved storage stability
	(significantly less supernatant and no sedimentation)
	+ significantly improved tensile strain at break
	+ higher impact strength

	Table of Resul	ts	HOFFMANN			
INTRODUCTION	Control Neuburg Siliceous Earth					
EXPERIMENTAL RESULTS				Sillitin Z 86 puriss	Silfit Z 91	Aktifit PF 115
SUMMARY <u>APPENDIX</u>	Viscosity Component A @ 0.1 s <sup>-1</sup> @ 1000 s <sup>-1</sup>	Pa·s Pa·s	5.4 3.8	6.4 5.1	10.7 5.8	35.9 5.4
	Sedimentation 3 months @ 40°C Supernatant Hard sediment	% %	44 5	6 3	8 5	9
	Hardness, 15 s	Shore D	81	81	80	81
	Tensile modulus	MPa	3340	3560	3560	3380
	Tensile strength	MPa	52.7	53.2	53.4	52.0
	Tensile strain at break	%	3.9	5.4	7.0	7.0
	Impact strength Charpy	kJ/m²	24	20	23	31
ESTATION ST	VM-0/0321/03.2021					



