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Approval: May 2008



VM / Dr. Alexander Risch

**Neuburg Siliceous Earth -  
Functional Filler  
Ways to Optimize  
Industrial Epoxy Flooring**

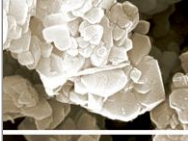


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Appendix: viscosity

# 1 Benefits and application areas

Industrial floorings are systems based on two-component epoxy formulations with extremely high load-bearing capacity.

	<h2>Benefits of Epoxy floorings</h2> 
<p>INDEX</p> <p><u>BENEFITS AND APPLICATION AREAS</u></p> <p>EXPERIMENTAL</p> <p>RESULTS</p> <p>SUMMARY</p>	<ul style="list-style-type: none"><li>• self-leveling properties</li><li>• application with no visible joints</li><li>• robustness</li><li>• high resistance against chemicals</li><li>• walking safety</li><li>• easy maintenance</li><li>• hygiene</li><li>• physiological harmlessness</li><li>• electric conductivity (with special formulations)</li></ul>
	

	<h2>Application areas for Epoxy floorings</h2> 
<p>INDEX</p> <p><u>BENEFITS AND APPLICATION AREAS</u></p> <p>EXPERIMENTAL</p> <p>RESULTS</p> <p>SUMMARY</p>	<ul style="list-style-type: none"><li>• Production floors and storerooms</li><li>• Sales and exposition rooms</li><li>• Exhibition halls</li><li>• Garages and parking decks</li><li>• Workshops</li><li>• Laboratories</li><li>• Cellar rooms</li></ul>
	

The present study aimed at evaluating Neuburg Siliceous Earth in pertinent formulations as a partial or total replacement of conventionally used fillers and additives. In so doing, the typical preparation and application methods were maintained, and the coatings were subjected to appropriate performance tests.

## 2 Experimental

### 2.1 Morphology and characteristic properties of the fine fillers used

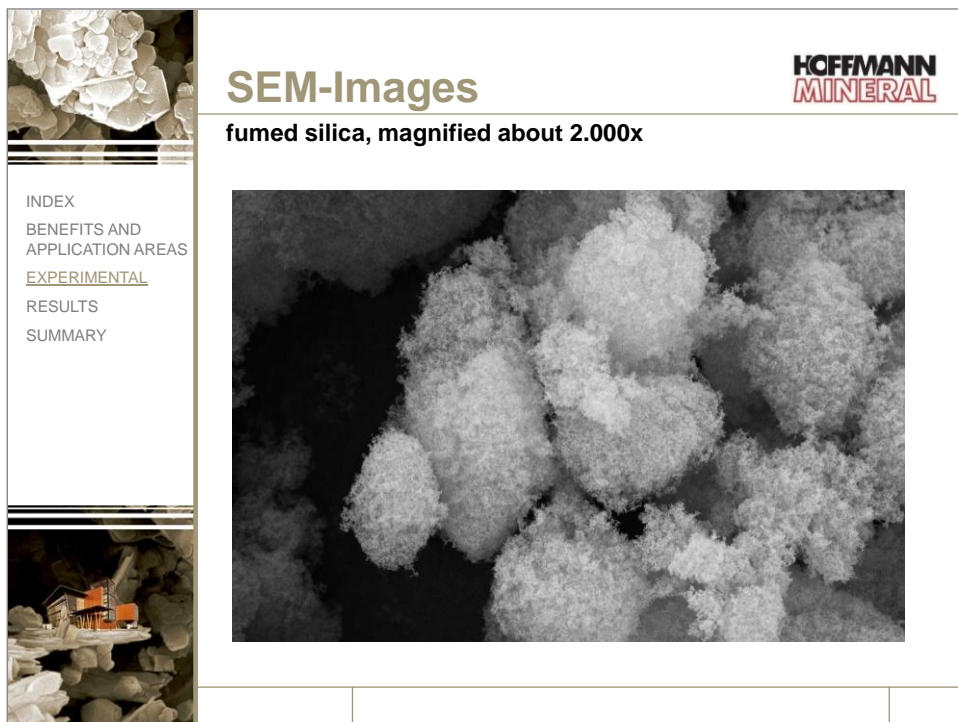
#### Barite

Barite (barium sulfate) is a filler characterized by a compact corpuscular grain shape. Along with its high density, this accounts for a low oil absorption and low specific surface area.



#### Hydrophobic (treated) fumed silica

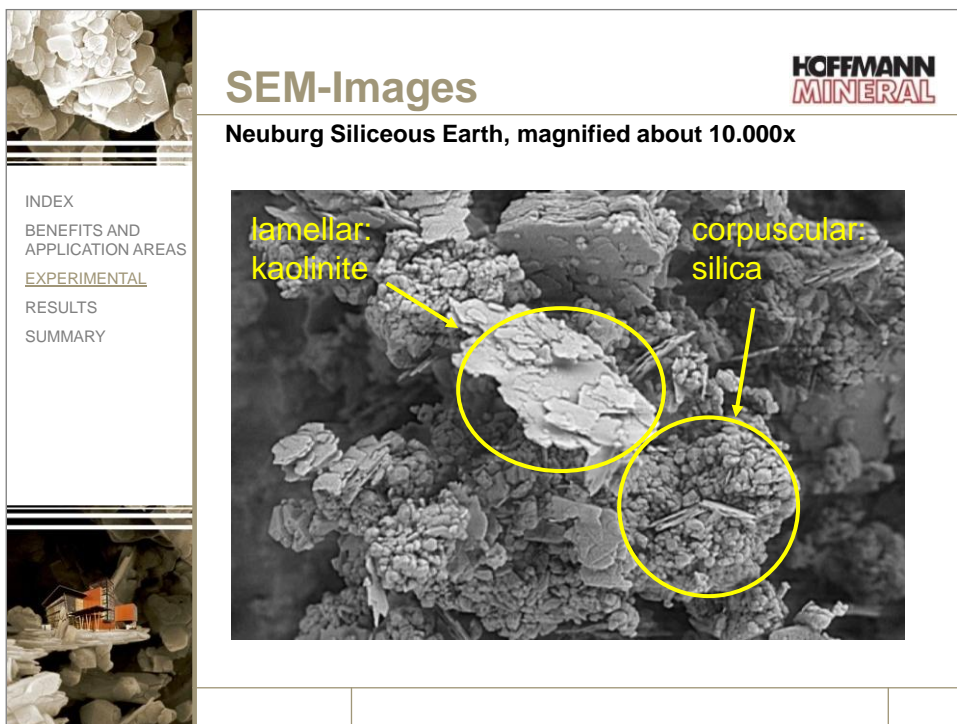
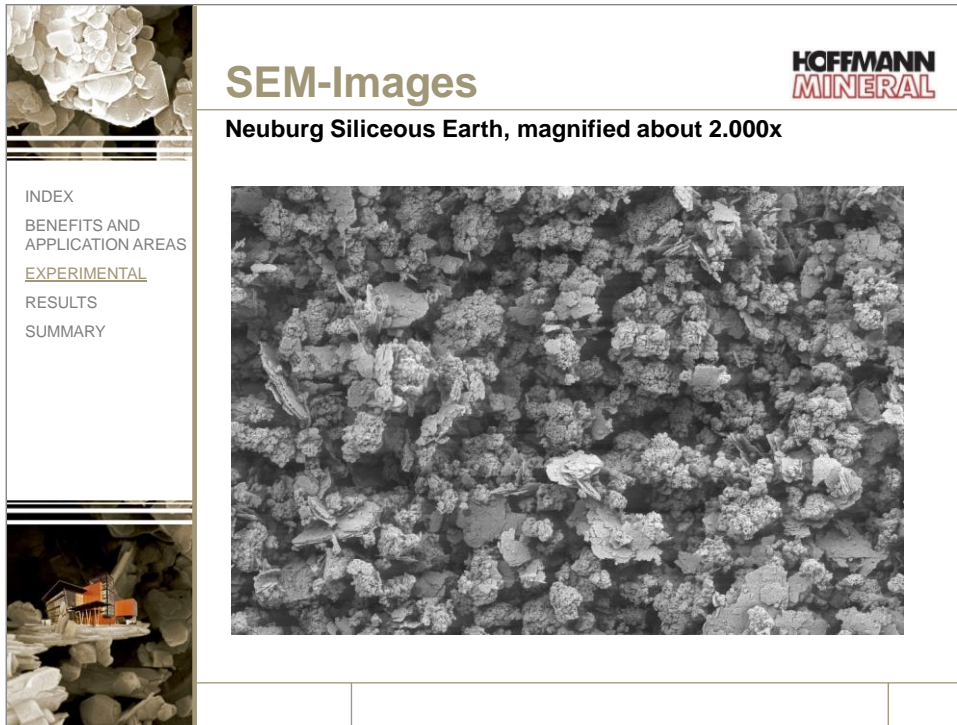
In view of its high specific surface area, hydrophobic fumed silica is used as a rheological additive. The primary particles, while being of a corpuscular nature, are present in the form of aggregates. These again assemble to form agglomerates, which can be broken down by suitable dispersion conditions.



## Neuburg Siliceous Earth

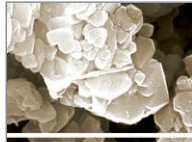
The Neuburg Siliceous Earth, extracted in the surrounding of Neuburg (Danube), is a natural combination of corpuscular, crypto-crystalline and amorphous silica and lamellar kaolinite: a loose mixture impossible to separate by physical methods. As a result of natural formation, the silica portion exhibits a round grain shape and consists of aggregated, crypto-crystalline primary particles of about 200 nm diameter, which are covered by amorphous silica opal-like. Such a structure is responsible for a relatively high specific surface area and oil absorption, which result, besides rheological activity, also in favorable mechanical properties.

The morphology of the Neuburg Siliceous Earth is depicted by the following pictures:



The table lists the characteristic properties of the functional fine fillers used:

	Reference		Neuburg Siliceous Earth (NSE)	
	Barite	fumed silica	Sillitin Z 86	Aktisil EM
Particle shape	corpuscular	corpuscular <i>aggregated</i>	lamellar / corpuscular <i>aggregated</i>	lamellar / corpuscular <i>aggregated</i>
Brightness Y	90		82	82
Brightness Z	97		77	77
Particle size distribution d50 [µm]	4.0		1.9	2.0
Particle size distribution d97 [µm]	17		7.6	9.4
Sieve residue > 40 µm [mg/kg]	230		6	4
Density [g/cm <sup>3</sup> ]	4.4		2.6	2.6
Oil absorption [g/100g]	13		49	50
Surface area [m <sup>2</sup> /g]	< 1	100	12	
Surface treatment	none	hydro- phobic	none	epoxy silane



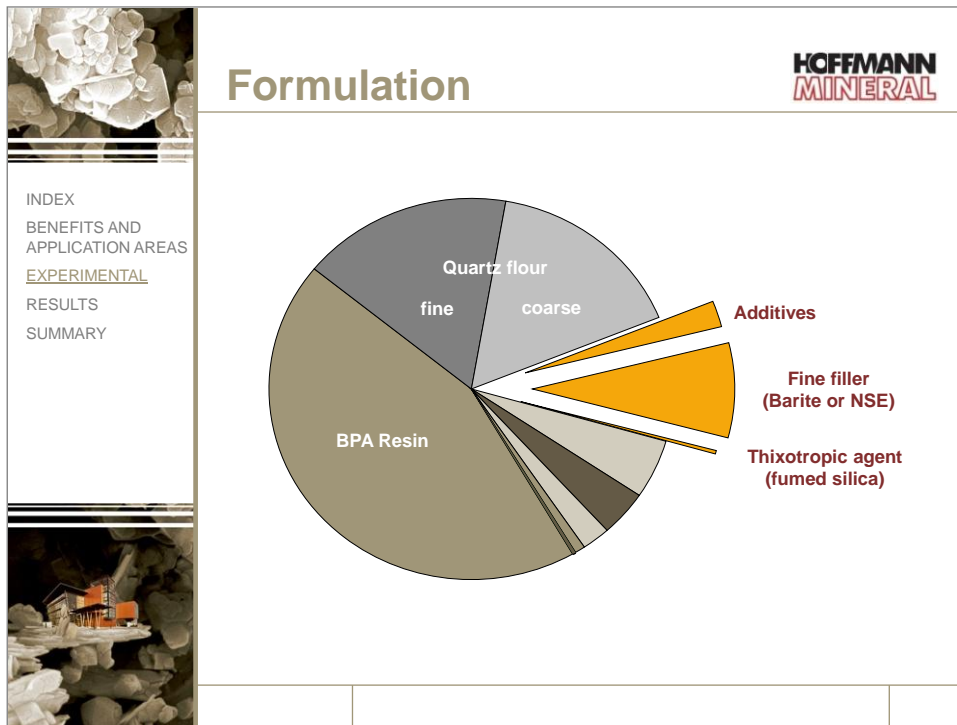
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## Filler characteristics

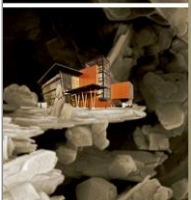
**HOFFMANN  
MINERAL**

## 2.2 Base formulation and test design






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### Formulation in detail



<u>Component A</u>	parts by weight (pbw)
<b>Bisphenol A epoxy resin (D.E.R. 336, DOW)</b>	<b>44.0</b>
<b>Defoamer</b>	<b>0.8</b>
<b>Quartz flour fine (d<sub>50</sub>/d<sub>95</sub>: 20/70µm)</b>	<b>17.0</b>
<b>Quartz flour coarse (d<sub>50</sub>/d<sub>95</sub>: 30/90µm)</b>	<b>16.0</b>
<b>Additives (leveling, dispersing, surfactant)</b>	<b>2.5 / 0</b>
<b>Fine filler (Barite or NSE)</b>	<b>8.0</b>
<b>Thixotropic agent (fumed silica)</b>	<b>0.3 / 0</b>
<b>Pigments (titanium dioxide and iron oxide types)</b>	<b>5.0</b>
<b>Reactive diluent (1,6-hexanediol diglycidylether)</b>	<b>4.0</b>
<b>Benzyl alcohol</b>	<b>2.0</b>
<b>Isopropanol</b>	<b>0.4</b>
<b>Total</b>	<b>100.0</b>

<u>Component B</u>	parts by weight (pbw)
<b>Hardener, based on isophoron diamine</b>	<b>20.0</b>

**Stoichiometric mixing ratio EP/amine is 1**

The tests with the different formulations were principally carried out in identical ways.

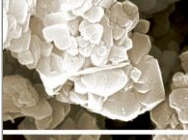
The A components were prepared in a laboratory dissolver. After introducing epoxy resin, defoamer and dispersion agent, the pigments and fillers were stirred in and dispersed with a peripheral speed of 15 m/s up to a batch temperature of 68 °C. After cooling down, the remaining ingredients were added.

The addition of the hardener for sample preparation was made by hand.


Compared to the base formulation, the following variations were tested:

The barite used as a fine filler in the base formulation was replaced with the same weight of Neuburg Siliceous Earth while simultaneously eliminating the treated fumed silica thixotrope. In view of the high density and the moderate rheological activity of the barite, there is no need for silica addition when working with Neuburg Siliceous Earth instead.


At the same time, the additives in the formulation were varied, i.e. reduced down just to the deaerating agent.



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## Design of Experiments



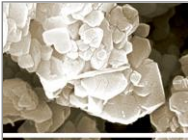
**Step 1**

**Replacement of Fine filler fraction and Thixotropic agent**


Barite	8 pbw	→	Sillitin Z 86	8 pbw
fumed silica	0.3 pbw			

**Variation of additives**


Defoamer	0.8 pbw	→	Dispersion additive 1b rest is the same
Dispersion add. 1a	0.5 pbw		
Dispersion add. 2	0.3 pbw		
Leveling additive	1.2 pbw		
Wetting agent	0.5 pbw		



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## Design of Experiments



**Step 2**

**Replacement of additives**

Defoamer	0.8 pbw	→	Defoamer 0.8 pbw	no other additives
Dispersion add. 1a	0.5 pbw			
Dispersion add. 2	0.3 pbw			
Leveling additive	1.2 pbw			
Wetting agent	0.5 pbw			

<b>Fine fillers:</b> Barite / fumed silica Sillitin Z 86 Aktisil EM Barite no fine filler
--

## **3 Results**

### **3.1 Processing properties**

The assessment of the processing properties included mixing a batch of A component and hardener, casting the mix onto a paperboard and distributing the mass over a defined area with a coarse notched trowel. 15 minutes later, a freshly mixed batch was applied adjacent to the first one, and distributed with a slight overlap. After another 5 minutes, half of the sample was rolled off with a spiked roller.

The dry layer thickness in all cases was about 2 mm.

The processing properties are markedly affected by the fillers, the wetting and the dispersing agents as well as by the other additives included in the base formulation. After complete cure, a visual assessment of the samples was carried out relative to different criteria.

#### **Processing properties upon addition of coarse quartz sand**

In actual practice, during mixing operations on site frequently coarse quartz sand is added in order to increase the layer thickness and/or save costs.

The processing properties of formulations without additives resp. with dispersion additive 1a were compared up to a sand content of 80 parts by weight (on 100 p.b.w. of formulation).

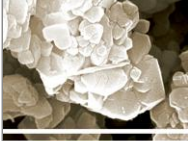
For the tests the following quartz sand types were used:

- Quarzsand F 36 (grain size approx. 0.1-0.36 mm), Quarzwerke GmbH
- Geba FG (grain size 0.03-0.6 mm), Gebrüder Dorfner GmbH & Co.


On principle, the workability of all formulations tested was negatively affected by the addition of coarse quartz sand, and this in proportion to the amount of sand added.

With equal sand additions, the following discussed differences between the fine fillers are largely maintained. The use of Neuburg Siliceous Earth leads to improved application properties also in formulations with sand additions, and Aktisil EM gives, compared to Sillitin Z 86, further benefits regarding deaeration and leveling.

## Leveling (surface smoothness, closing of the notched trowel)




### Assessment of Processing



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#### Leveling



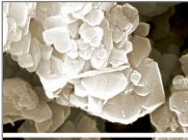
**wavy surface**




**smooth surface**



On principle, the compounds with the barite/treated silica blend offer slight advantages in leveling because of the lower volume filler proportion. When eliminating the additives, how-ever, the leveling properties heavily change in favor of the Sillitin Z 86 or Aktisil EM batch.




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#### Leveling


Step 1	
Barite + fumed silica / Dispersion additive 1a	+
Sillitin Z 86 / Dispersion additive 1a	-
Barite + fumed silica / Dispersion additive 1b	+
Sillitin Z 86 / Dispersion additive 1b	o
Step 2	
Barite + fumed silica / no additives	+
Sillitin Z 86 / no additives	++
Aktisil EM / no additives	++
Barite / no additives	++
no fine filler / no additives	++




## Deaeration (air inclusions and/or craters from burst blisters)




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### Assessment of Processing



#### Deaeration

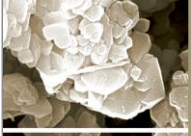


**entrapped air bubbles  
and craters**




**no air bubbles  
or craters**


Shortcomings are particularly evident with the treated silica formulations. The largest differences come out in the additive-free systems. The barite/silica combination, in contrast to Sillitin Z 86, here leads by far to the poorest deaeration performance.



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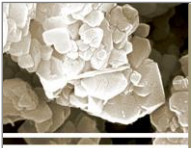
### Assessment of Processing



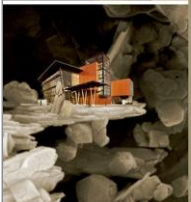
#### Deaeration

Step 1	
Barite + fumed silica / Dispersion additive 1a	-- --
Sillitin Z 86 / Dispersion additive 1a	--
Barite + fumed silica / Dispersion additive 1b	--
Sillitin Z 86 / Dispersion additive 1b	++
Step 2	
Barite + fumed silica / no additives	-- --
Sillitin Z 86 / no additives	+
Aktisil EM / no additives	○
Barite / no additives	++
no fine filler / no additives	++

**Pigment stability (homogenous coloring, flooding of pigments, formation of Bénard cells)**

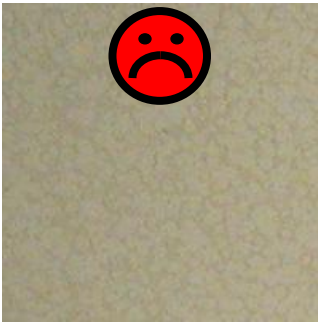


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### Pigment stability

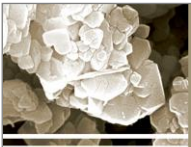


irregular color,  
Bénard cells

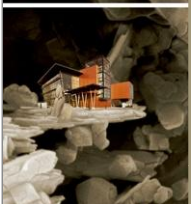


evenly colored

The formulations containing Neuburg Siliceous Earth, especially Sillitin Z 86, offer a more “uniform” visual impression compared to the systems without filler resp. with barite.



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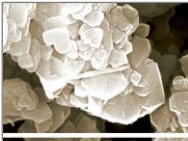


## Assessment of Processing HOFFMANN MINERAL

### Pigment stability

Step 1	
Barite + fumed silica / Dispersion additive 1a	-- --
Sillitin Z 86 / Dispersion additive 1a	○
Barite + fumed silica / Dispersion additive 1b	-- --
Sillitin Z 86 / Dispersion additive 1b	○
Step 2	
Barite + fumed silica / no additives	-
Sillitin Z 86 / no additives	+
Aktisil EM / no additives	○
Barite / no additives	-
no fine filler / no additives	○

**Appearance of crossover area (color and structure changes at the joint between subsequent batches)**



**Assessment of Processing** **HOFFMANN MINERAL**

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**Appearance of crossover area**




**irregular color,  
and / or wavy surface**




**evenly colored,  
smooth surface**

---



The visible transition of adjacent, subsequent batches is more pronounced, even after working with the spiked roller, for the barite compared with the Sillitin formulations. The best results were obtained with the additive-free Sillitin Z 86 compound, where no color or structure changes could be observed in the crossover area.



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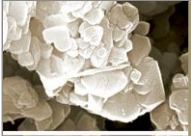
**Appearance of crossover area**

Step 1	
Barite + fumed silica / Dispersion additive 1a	-
Sillitin Z 86 / Dispersion additive 1a	○
Barite + fumed silica / Dispersion additive 1b	-
Sillitin Z 86 / Dispersion additive 1b	○
Step 2	
Barite + fumed silica / no additives	○
Sillitin Z 86 / no additives	+
Aktisil EM / no additives	○
Barite / no additives	-
no fine filler / no additives	○

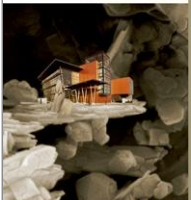
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### 3.2 Storage stability A-component (separation, sedimentation)




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## Assessment of storage stability

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### Sedimentation component A

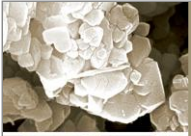


**colored separation and / or hard sediment**




**no or only clear separation, no sediment**

The samples were stored for 10 weeks at 40 °C, which would correspond in actual fact to a storage time of 18 months at room temperature. With the systems tested, differences were only observed with respect to the amount of clear supernatant liquid, while no sediment was formed in any case. With the same additives, Neuburg Siliceous Earth imparts less tendency towards separation than barite. The largest amount of separation was found for the fine filler free formulation.



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## Assessment of storage stability

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### Sedimentation component A (10 weeks 40°C)

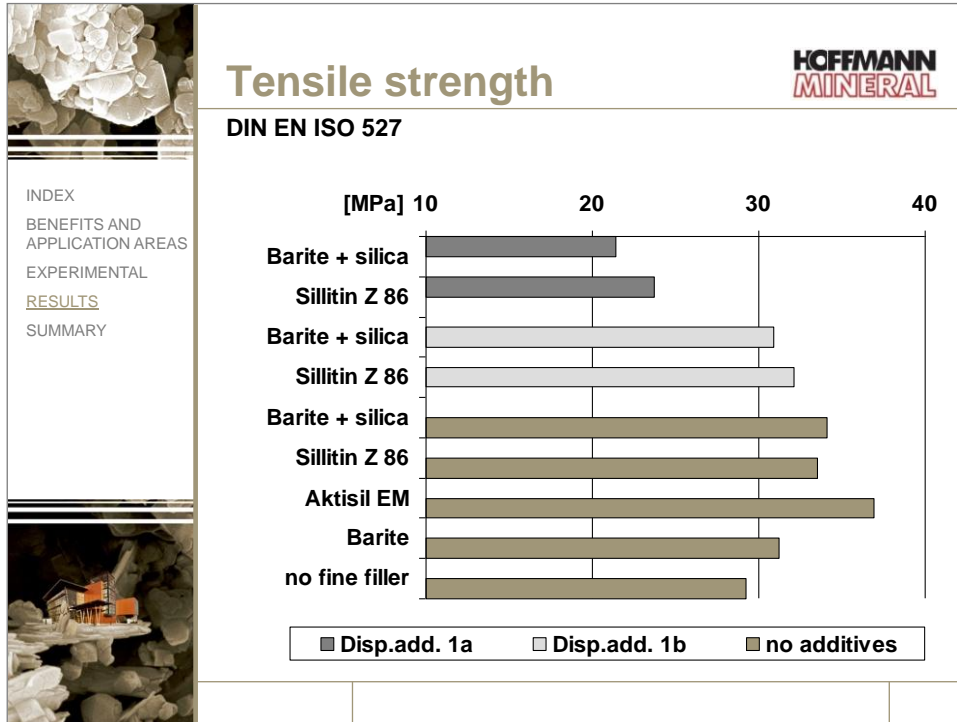
Step 1	
Barite + fumed silica / Dispersion additive 1a	+
Sillitin Z 86 / Dispersion additive 1a	+
Barite + fumed silica / Dispersion additive 1b	○
Sillitin Z 86 / Dispersion additive 1b	+
Step 2	
Barite + fumed silica / no additives	○
Sillitin Z 86 / no additives	+
Aktisil EM / no additives	+
Barite / no additives	○
no fine filler / no additives	-

### 3.3 Mechanical properties

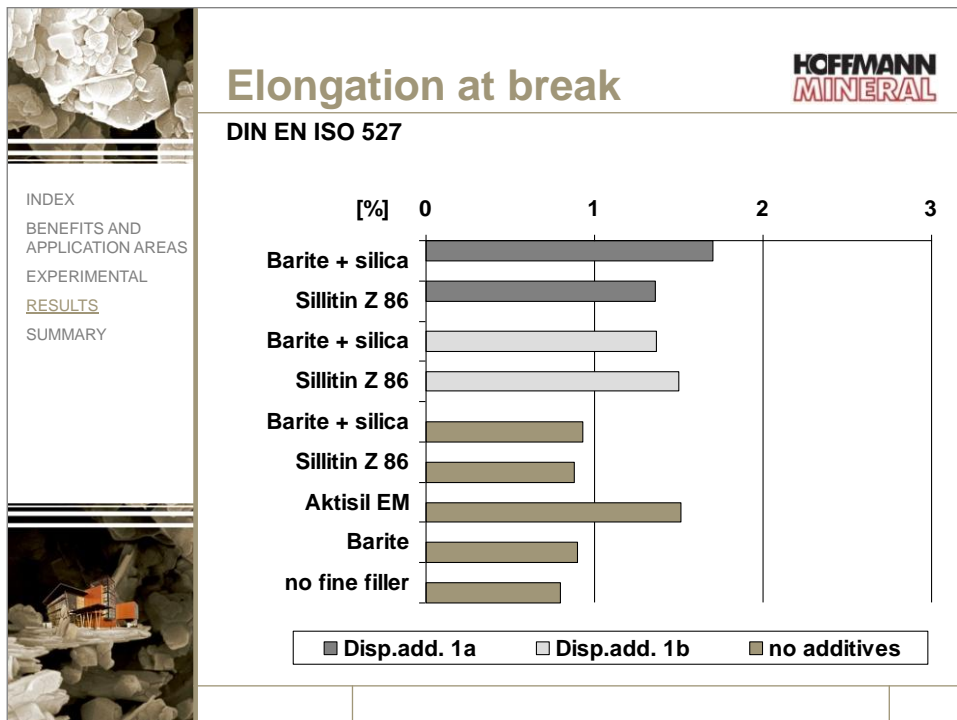
The required samples were prepared in appropriate casting molds, and were tested after complete cure (14 days at standard conditions, i.e. 23 °C and 50 % relative humidity).

#### Tensile test (DIN EN ISO 527)

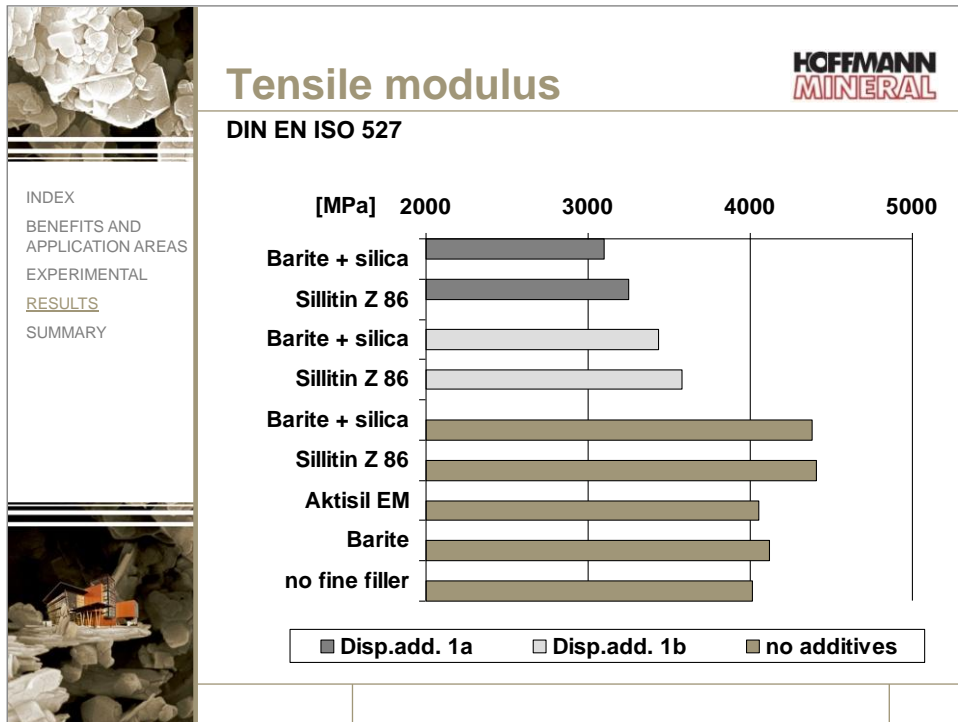
When working with the same additives, there is no significant difference between Neuburg Siliceous Earth and the barite compounds. Dispersion additive 1b or no additives at all will give markedly higher tensile strength compared with the base formulation.



Elongations at break come out at a similar level for Sillitin Z 86 and barite. Aktisil EM will lead to comparatively higher elongations.

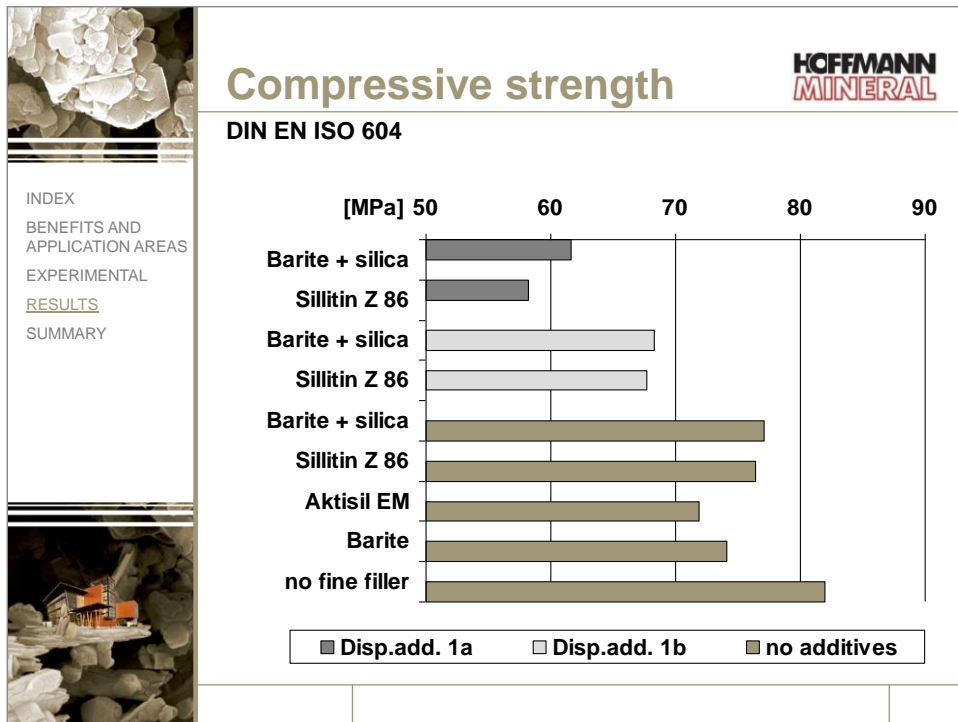


The tensile modulus too indicates a strong dependence on the additives used, while the fine filler containing compounds without additive exhibit the highest stiffness levels.



### Compression test (DIN EN ISO 604)

The systems formulated without additives show significantly higher compressive strength, with no big differences between barite and Neuburg Siliceous Earth, but the high level of the fine filler free formulation remains unequalled.



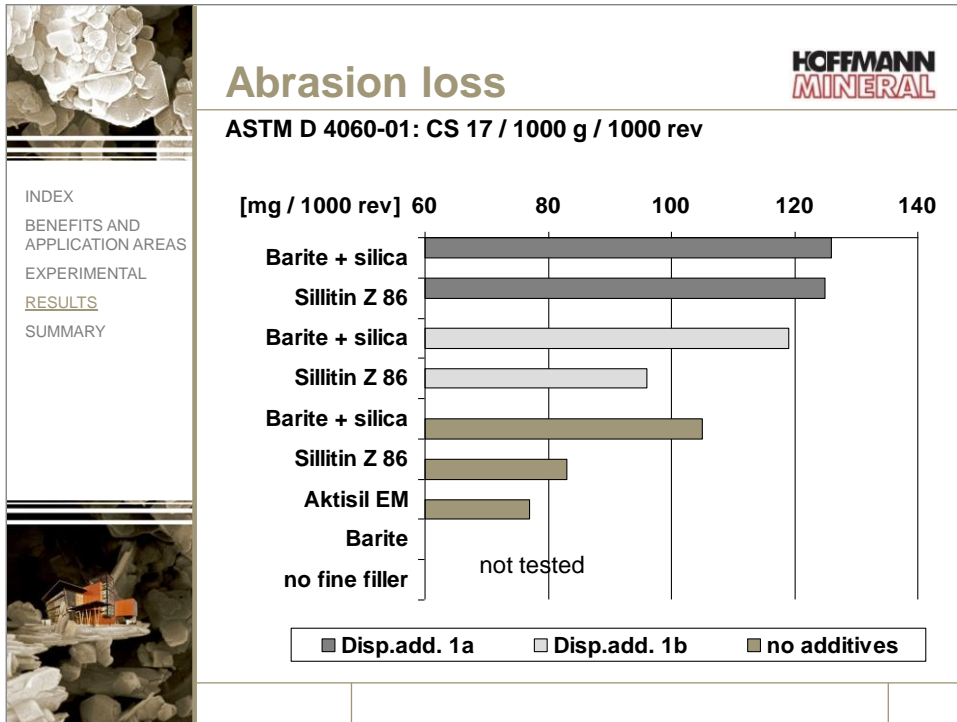
### Hardness Shore D (DIN EN ISO 868)

Reaching after 15 s: the hardness of the cured compounds was in the range of  $80 \pm 3$  Shore D.

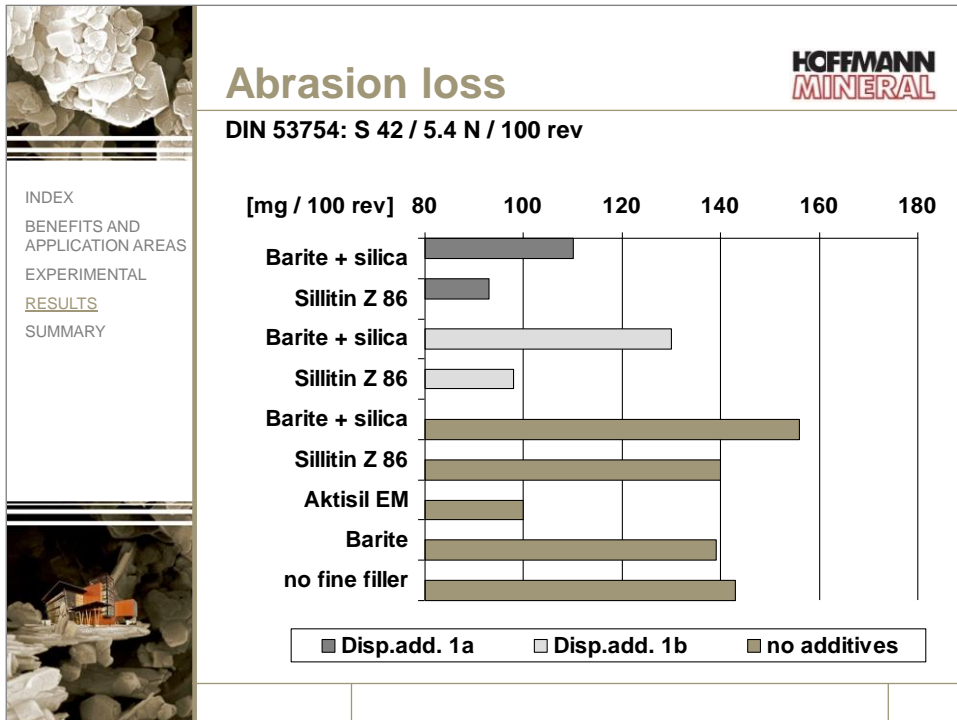
## Abrasion loss according to DIN 53754 resp. ASTM 4060-01

In direct comparison with barite/silica, Neuburg Siliceous Earth imparts about 20 % better abrasion figures.

Against fine abrasives (CS 17), the lowest abrasion loss is obtained with the additive-free compounds formulated with Neuburg Siliceous Earth fillers.

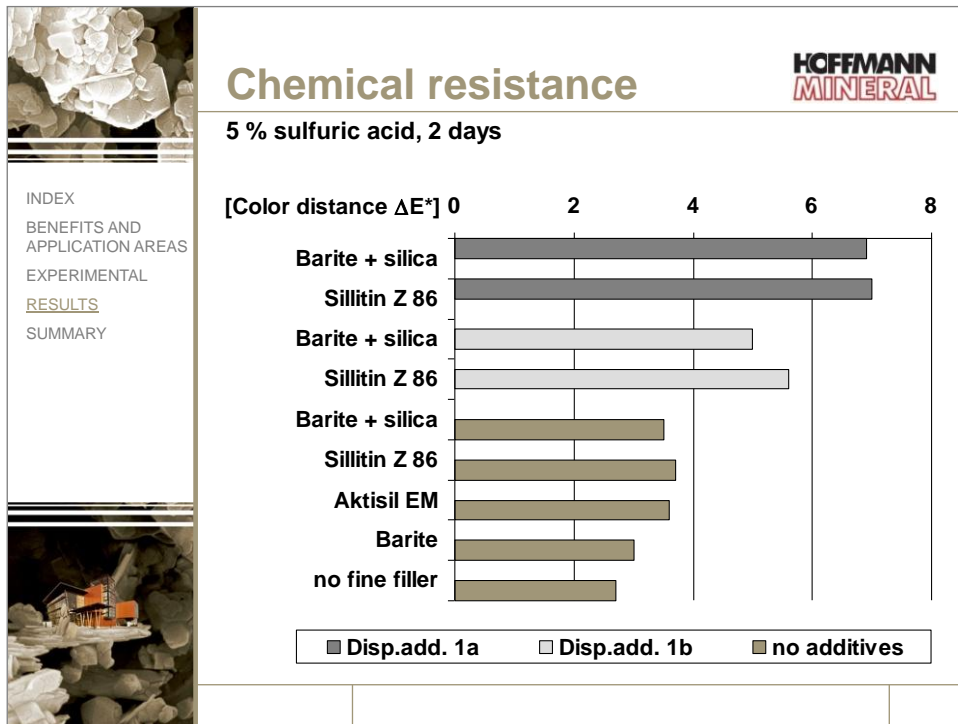


Against coarse abrasives (S 42), wear resistance falls off when additives are left out. Aktisil EM allows bringing the results back to the original level.



### 3.4 Chemical resistance

Resistance against chemicals is more distinctly influenced by additives than by the fillers incorporated. Depending on the exposure conditions, as a result the chemical resistance can be markedly improved with additive-free compounds.



The chemical resistance was also tested in 25 % sulfuric acid (24 h) as well as in red wine (8 weeks). The individual compounds showed only minor differences between each other.

## 4 Summary and conclusions

Processing and mechanical properties are markedly affected by the additives used. Neuburg Siliceous Earth, compared to the barite/silica blend, in epoxy based industrial flooring compounds offers advantages with respect to

- processing properties, in particular pigment stability and appearance of crossover area
- abrasion resistance.

In view of the improved processing properties, additives can be largely eliminated from the formulations, which would lead to additional cost savings in raw materials.

Sillitin Z 86, therefore, offers ways to combine the excellent mechanical properties of additive free formulations with outstanding processing properties under favorable cost aspects, and thus to markedly boost the performance of such flooring systems.

If high elongation at break and good wear resistance are primary requirements, Aktisil EM appears the material of choice.

The improved processing properties are also maintained in sand extended formulations. Aktisil EM here offers additional benefits with respect to deaeration and leveling.

For formulations without coarse fillers the use of Sillitin Z 86 puriss is recommended due to its excellent dispersing properties

*Our technical service suggestions and the information contained in this report are based on experience and are made to the best of our knowledge and belief, but must nevertheless be regarded as non-binding advice subject to no guarantee. Working and employment conditions over which we have no control exclude any damage claims arising from the use of our data and recommendations. Furthermore, we cannot assume any responsibility for any patent infringements which might result from the use of our information.*

# Appendix: viscosity

