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Unternehmensgruppe Hoffmann

Neuburg Siliceous Earth

for self-leveling

epoxy industrial flooring

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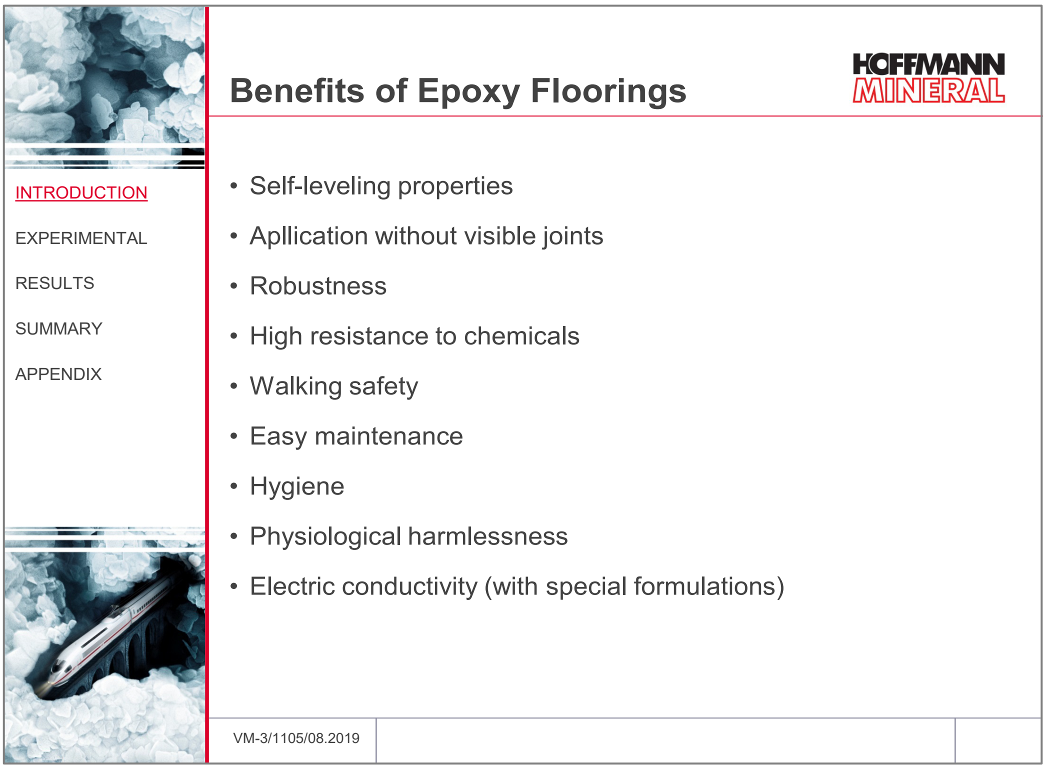
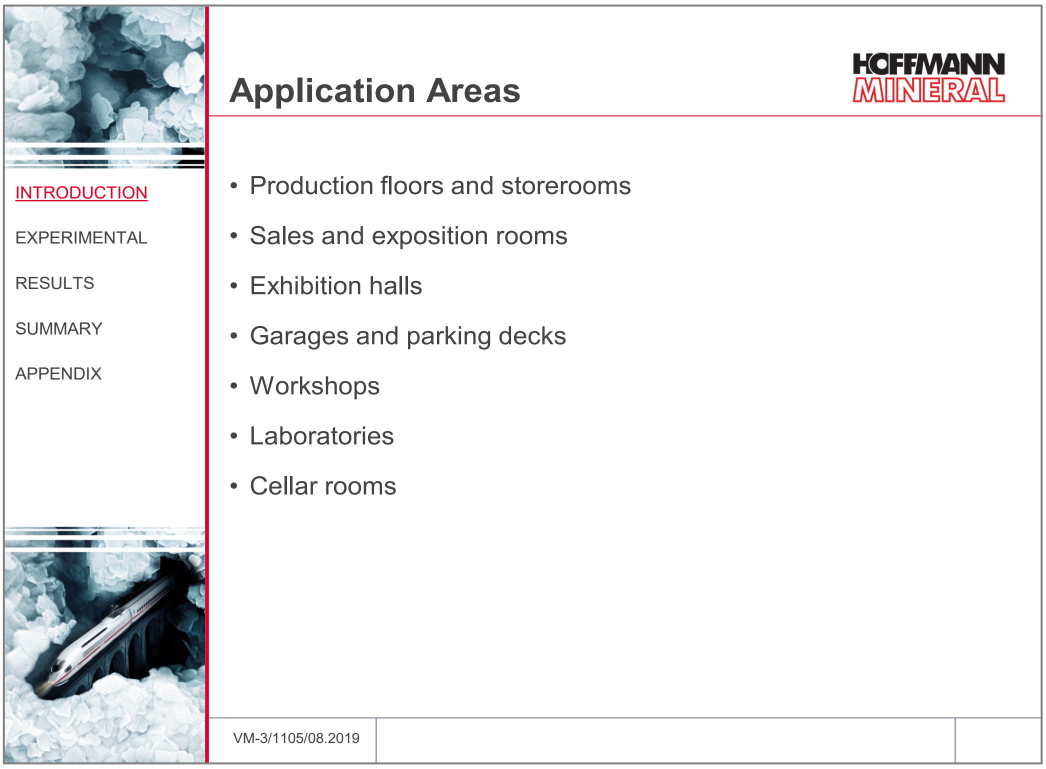
1 Benefits and application areas

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





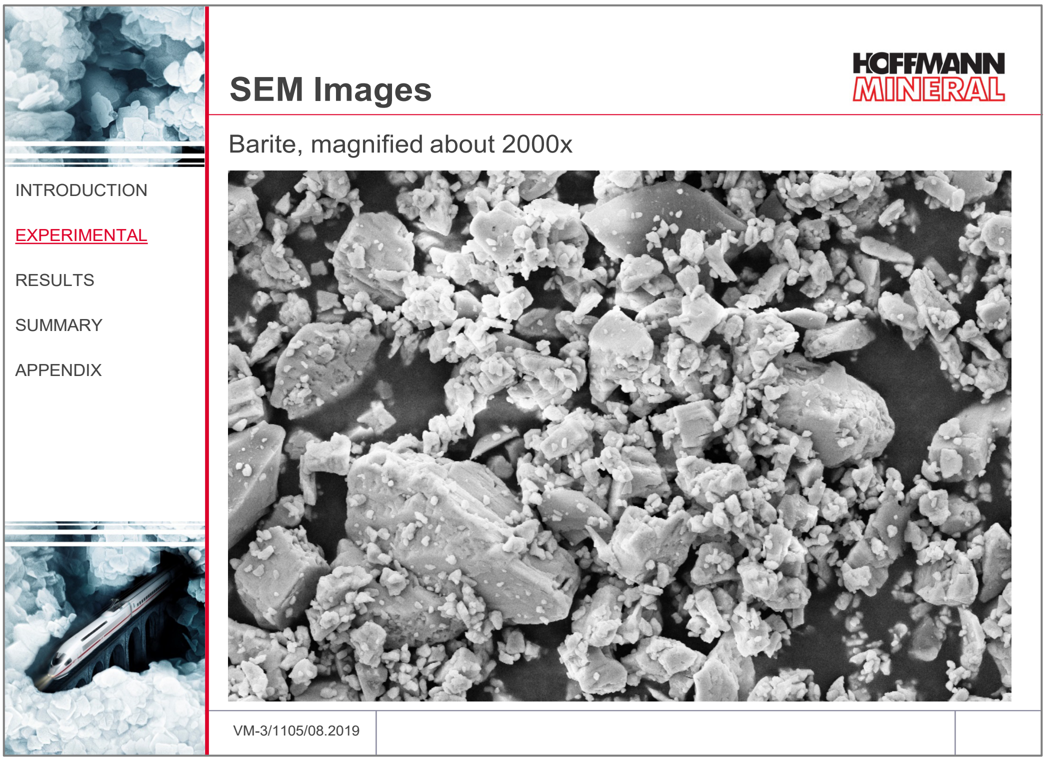
The present study aimed at evaluating Neuburg Siliceous Earth in pertinent formulations as a partial or total replacement of conventionally used fillers and additives. In so doing, the typical preparation and application methods were maintained, and the coatings were subjected to appropriate performance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

2 Experimental

2.1 Morphology and characteristic properties of the fine fillers used

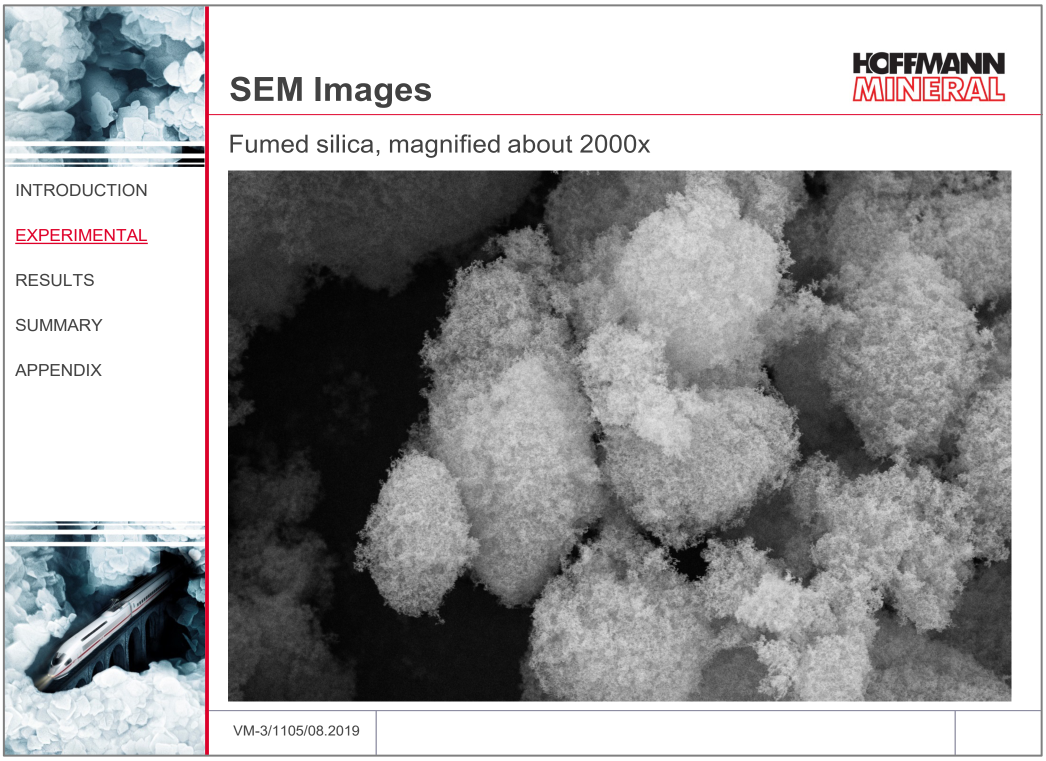
Barite

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

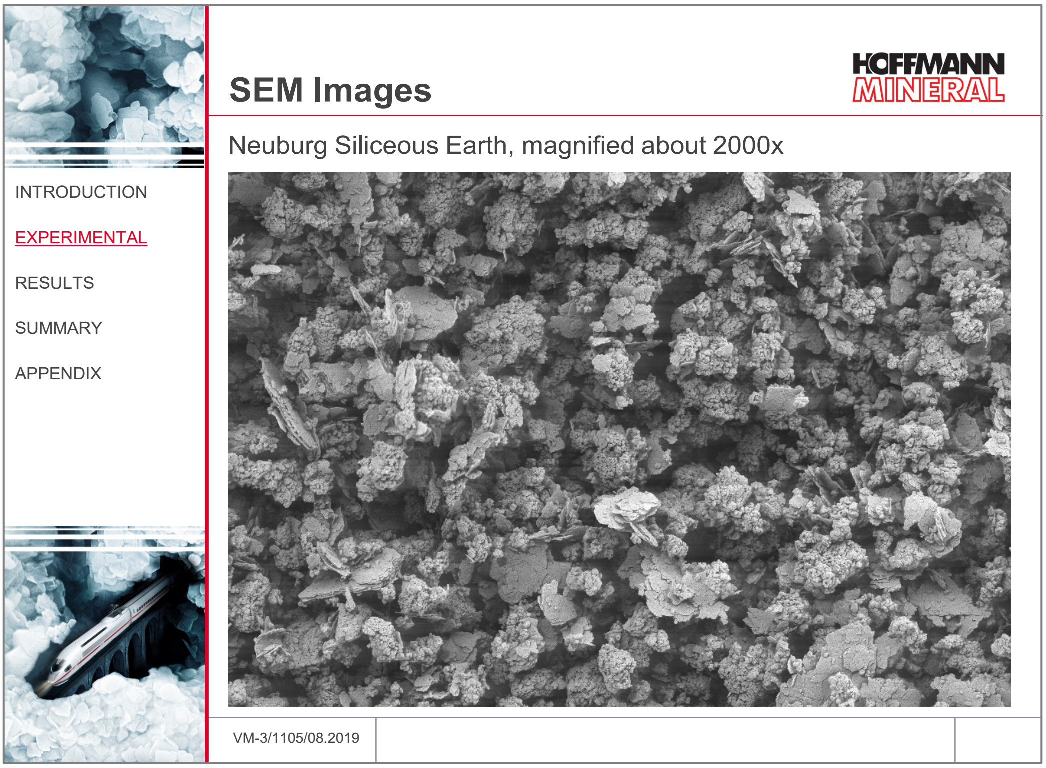
In view of its high specific surface area, hydrophobic fumed silica is used as a rheological additive. The primary particles, while being of a corpuscular nature, are present in the form of aggregates. These again assemble to form agglomerates, which can be broken down by suitable dispersion conditions.

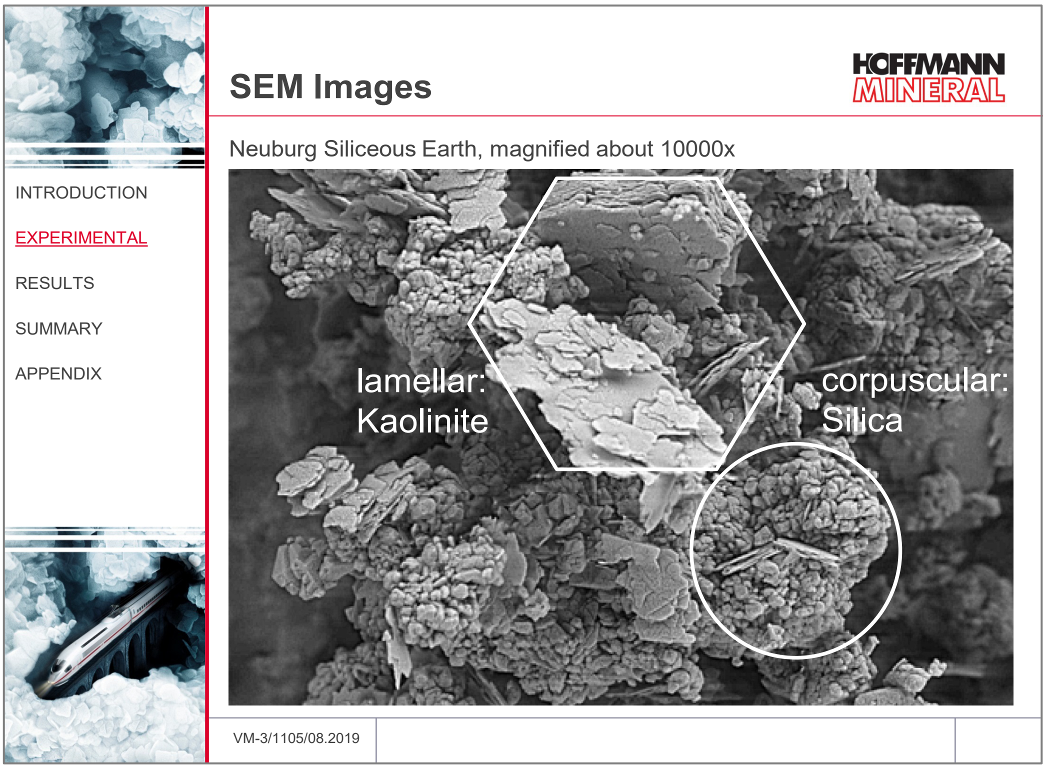


Neuburg Siliceous Earth

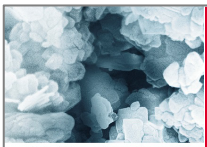
The Neuburg Siliceous Earth, extracted in the surrounding of Neuburg (Danube), is a natural combination of corpuscular Neuburg silica and lamellar kaolinite: a loose mixture impossible to separate by physical methods. As a result of natural aging, the silica portion exhibits a round grain shape and consists of aggregated cryptocrystalline primary particles of about 200 nm diameter. Such a structure is responsible for a relatively high specific surface area and oil absorption, which result, besides rheological activity, also in a whole range of application 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:



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Filler Characteristics

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	Reference		Neuburg Siliceous Earth (NSE)
	Barite	Fumed silica	Sillitin Z 86
Particle shape	corpuscular	corpuscular aggregated	lamellar / corpuscular aggregated
Brightness Y	90		82
Brightness Z	97		77
Particle size d_{50} [µm]	4.0		1.9
Particle size d_{97} [µm]	17		7.6
Sieve residue >40µm [mg/kg]	230		6
Density [g/cm³]	4.4		2.6
Oil absorption [g/100g]	13		49
BET surface area [m²/g]	<1	100	12
Surface treatment	none	hydro-phobic	none

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2.2 Base formulation and test design

Base Formulation

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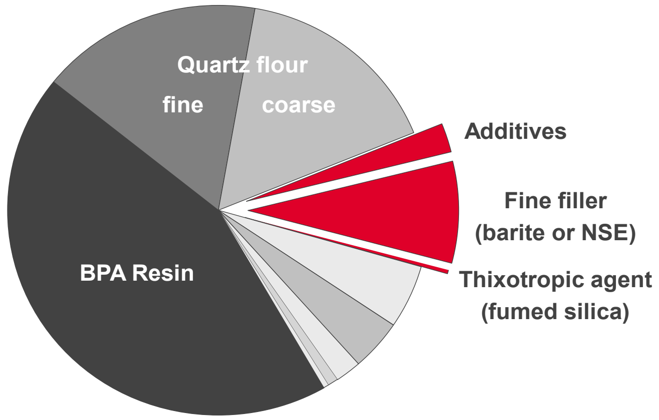

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Formulation in Detail

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

Component B		
Hardener, based on isophoron diamine	20.0	
Stoichiometric mixing ratio EP/amine is 1		

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

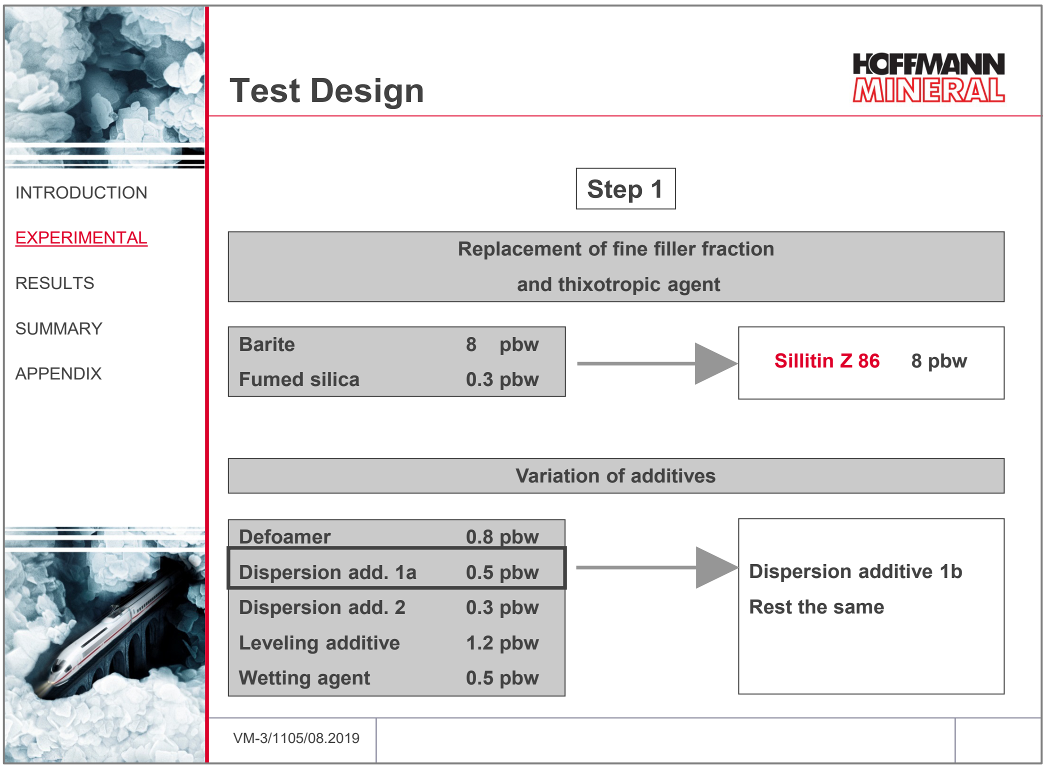
The A components were prepared in a laboratory dissolver. After introducing epoxy resin, defoamer and dispersion agent, the pigments and fillers were stirred in and dispersed with a peripheral speed of 15 m/s up to a batch temperature of 68 °C. After cooling down, the remaining ingredients were added.

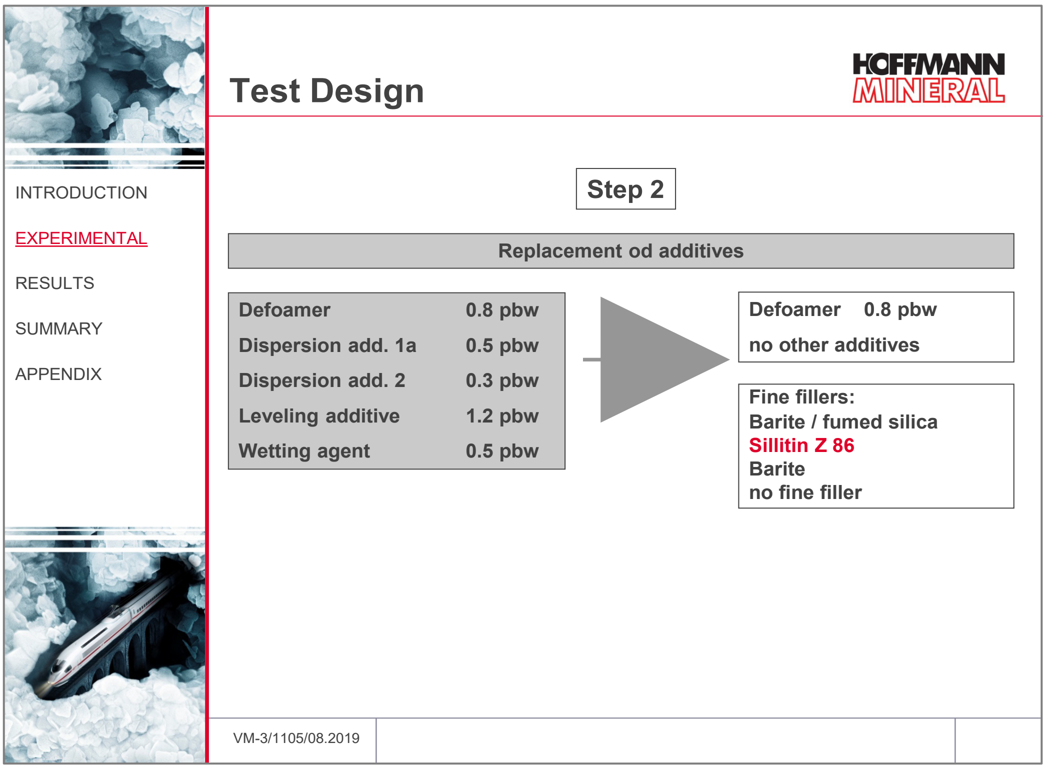
The addition of the hardener for sample preparation was made by hand.

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.





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

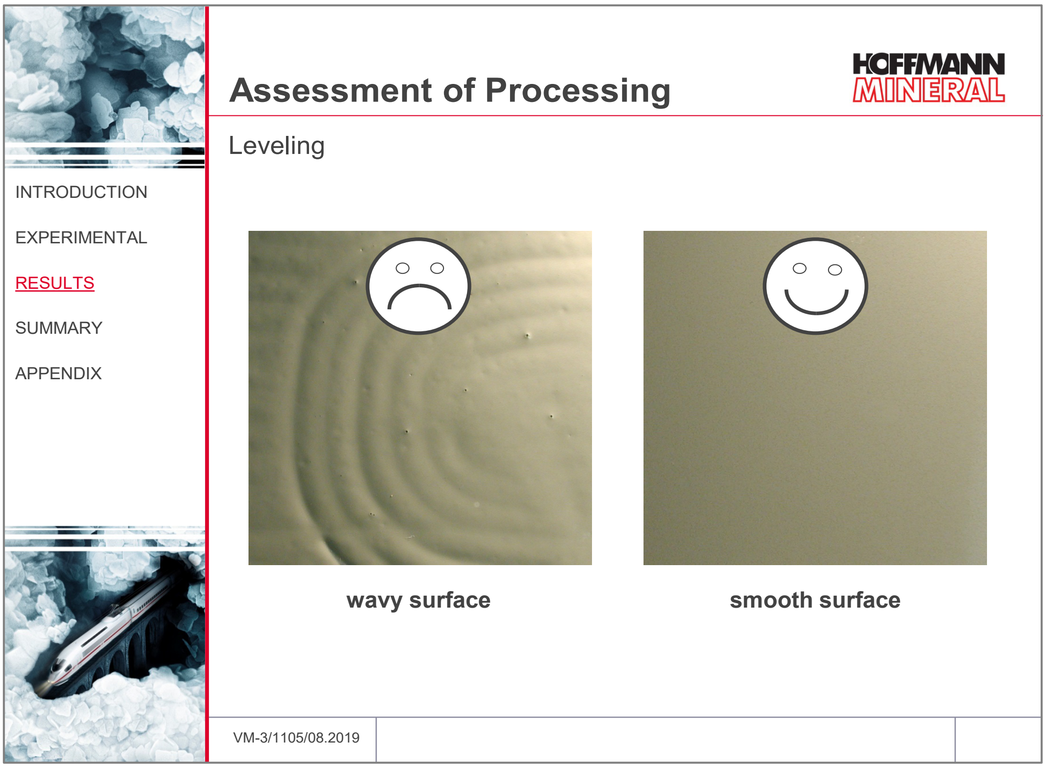
In actual practice, during mixing operations on site frequently coarse quartz sand is added in order to increase the layer thickness and/or save costs.
The processing properties of formulations without additives resp. with dispersion additive 1a were compared up to a sand content of 80 parts by weight (on 100 p.b.w. total formulation incl. hardener).

For the tests the following quartz sand types were used:

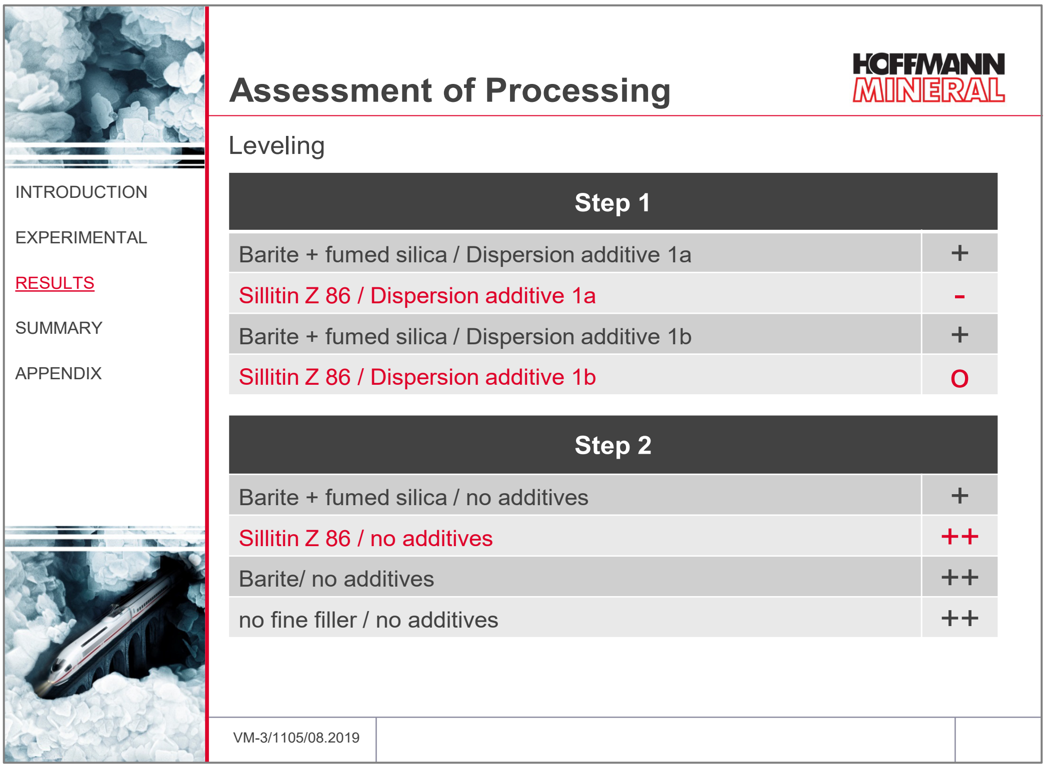
- Quarzsand F 36 (grain size approx. 0.1-0.36 mm), Quarzwerke GmbH
- Geba FG (grain size 0.03-0.6 mm), Gebrüder Dorfner GmbH & Co

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 addition.

Leveling (surface smoothness, closing of the notched trowel)



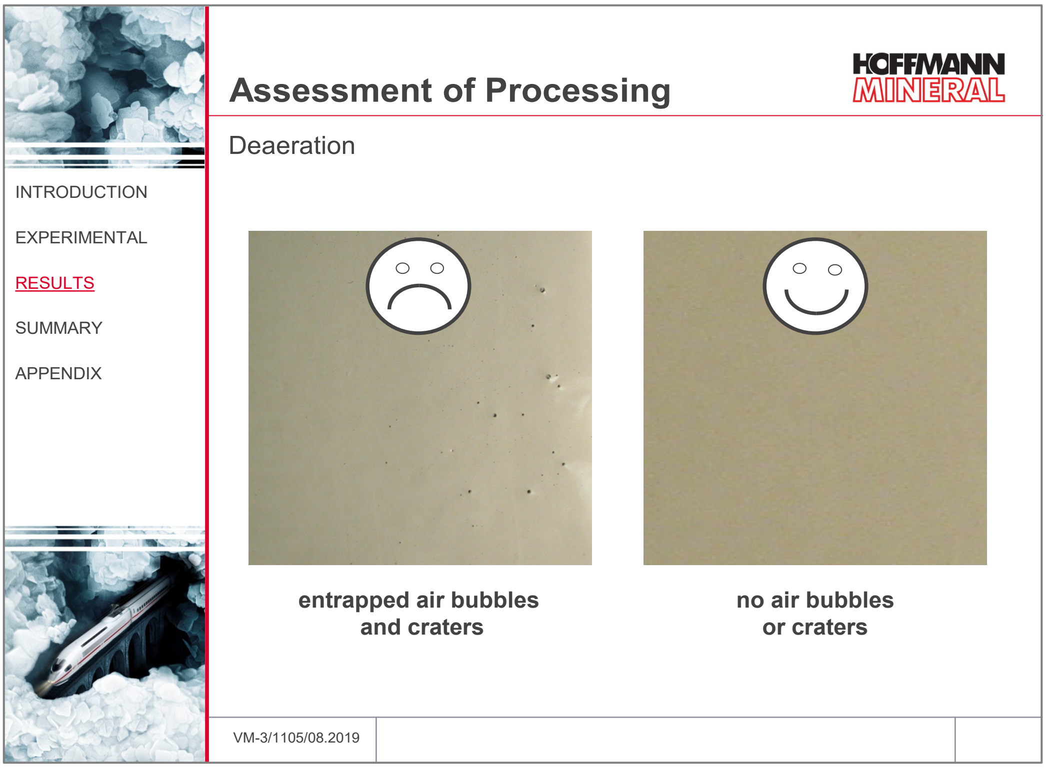
On principle, the compounds with the barite/treated silica blend offer slight advantages in leveling because of the lower volume filler proportion. When eliminating the additives, how-ever, the leveling properties heavily change in favor of the Sillitin Z 86 batch.



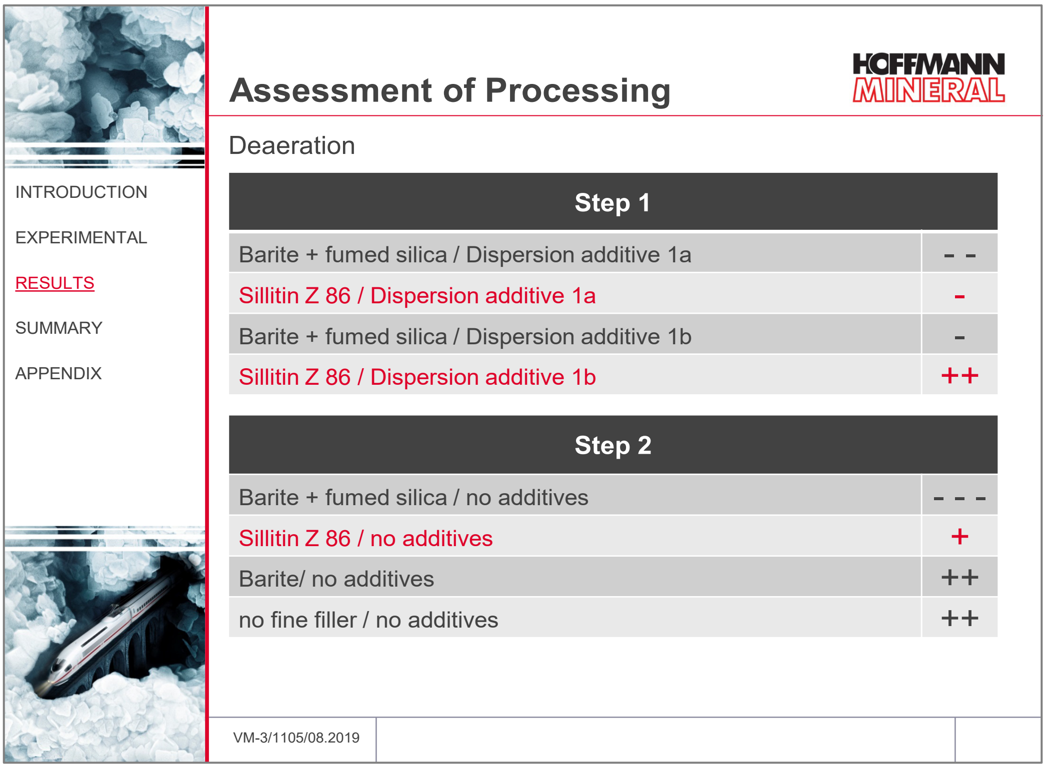
Step 1	
Barite + fumed silica / Dispersion additive 1a	+
Sillitin Z 86 / Dispersion additive 1a	-
Barite + fumed silica / Dispersion additive 1b	+
Sillitin Z 86 / Dispersion additive 1b	O

Step 2	
Barite + fumed silica / no additives	+
Sillitin Z 86 / no additives	++
Barite/ no additives	++
no fine filler / no additives	++

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



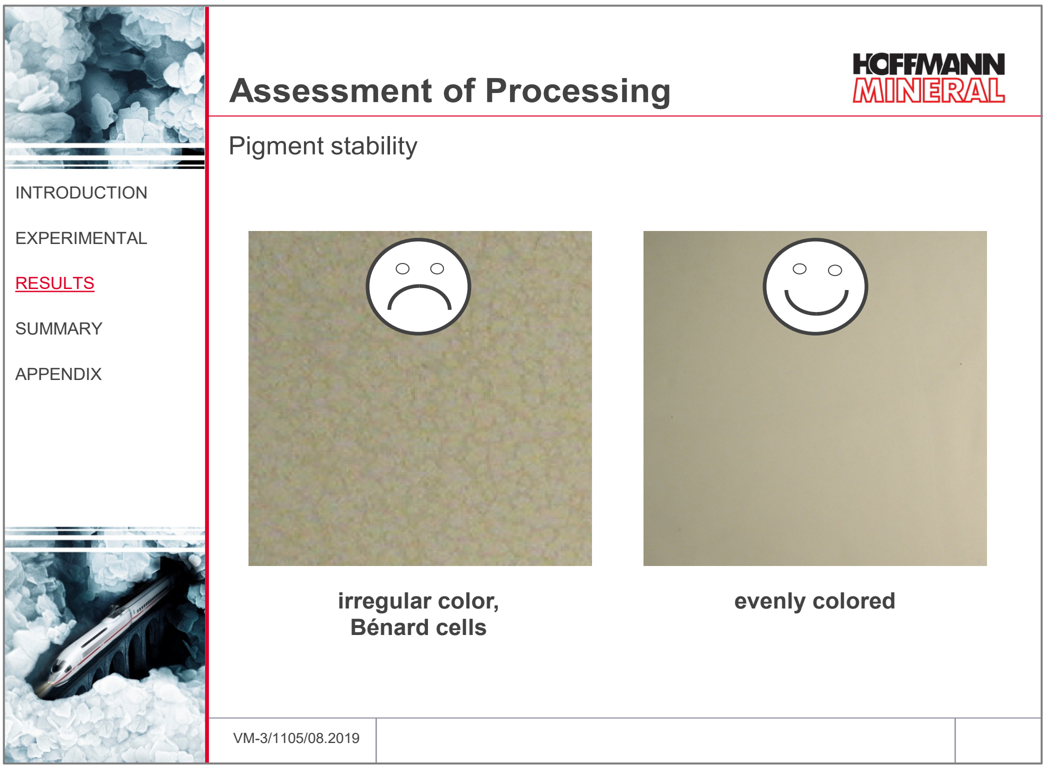
Shortcomings are particularly evident with the treated silica formulations. The largest differences come out in the additive-free systems. The barite/silica combination, in contrast to Sillitin Z 86, here leads by far to the poorest deaeration performance.



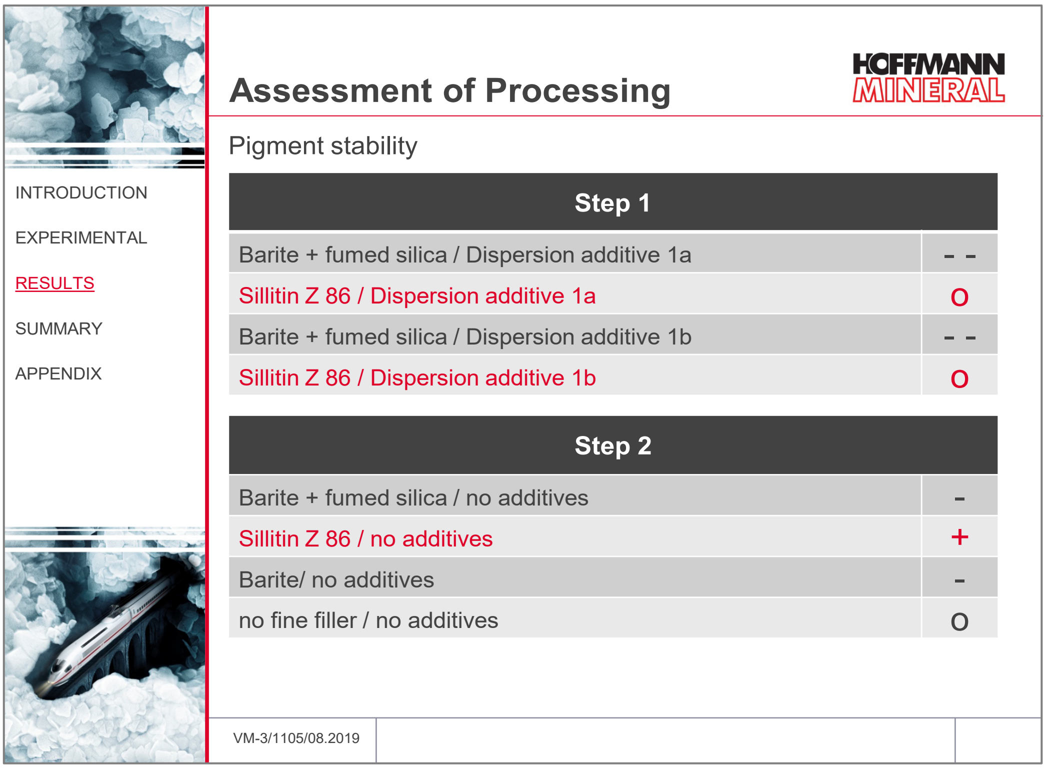
Step 1	
Barite + fumed silica / Dispersion additive 1a	- -
Sillitin Z 86 / Dispersion additive 1a	-
Barite + fumed silica / Dispersion additive 1b	-
Sillitin Z 86 / Dispersion additive 1b	++

Step 2	
Barite + fumed silica / no additives	- - -
Sillitin Z 86 / no additives	+
Barite/ no additives	++
no fine filler / no additives	++

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



The formulations containing Sillitin Z 86 offer a more "uniform" visual impression compared to the systems without filler resp. with barite.



Step 1	
Barite + fumed silica / Dispersion additive 1a	- -
Sillitin Z 86 / Dispersion additive 1a	O
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	+
Barite/ no additives	-
no fine filler / no additives	O

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

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Assessment of Processing

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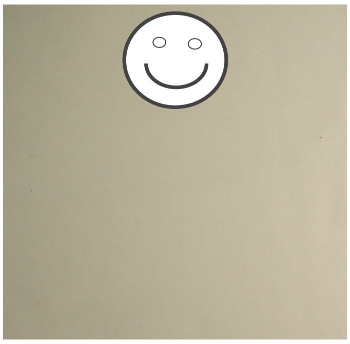
Appearance of crossover area

irregular color,
and / or wavy surface



evenly colored,
smooth surface

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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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Assessment of Processing

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Appearance of crossover area

Step 1	
Barite + fumed silica / Dispersion additive 1a	-
Sillitin Z 86 / Dispersion additive 1a	O
Barite + fumed silica / Dispersion additive 1b	-
Sillitin Z 86 / Dispersion additive 1b	O

Step 2	
Barite + fumed silica / no additives	O
Sillitin Z 86 / no additives	+
Barite/ no additives	-
no fine filler / no additives	O

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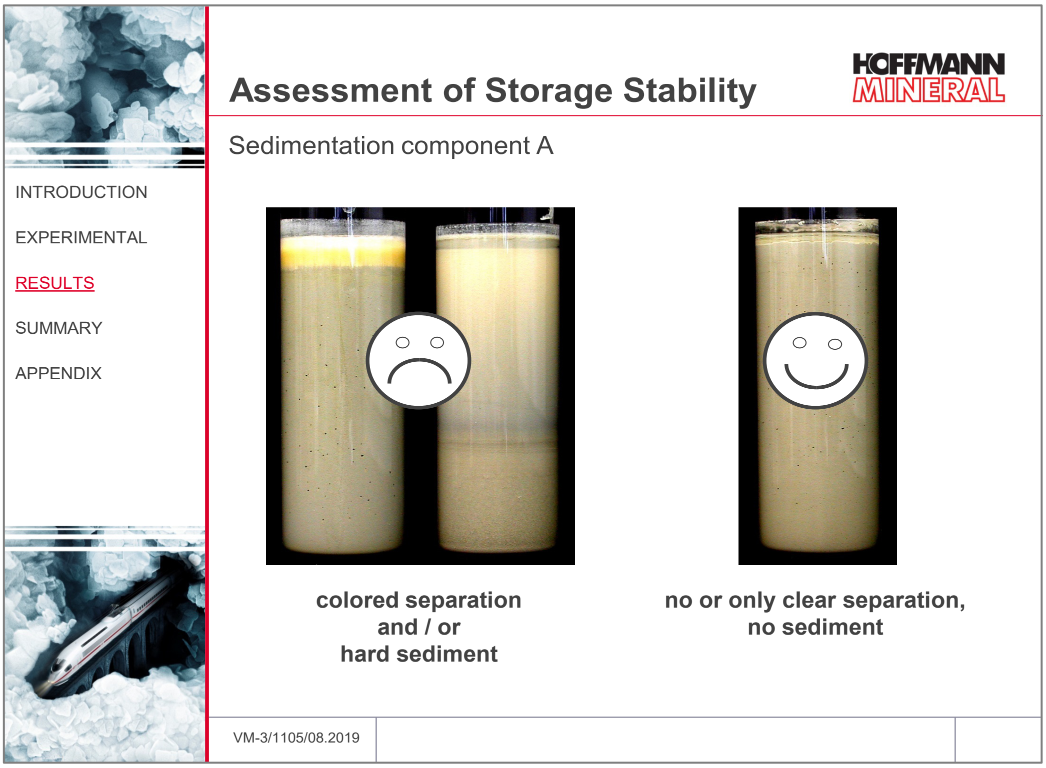
3.2 Storage stability A-component (separation, sedimentation)



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.

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	O
Sillitin Z 86 / Dispersion additive 1b	+

Step 2	
Barite + fumed silica / no additives	O
Sillitin Z 86 / no additives	+
Barite/ no additives	O
no fine filler / no additives	-

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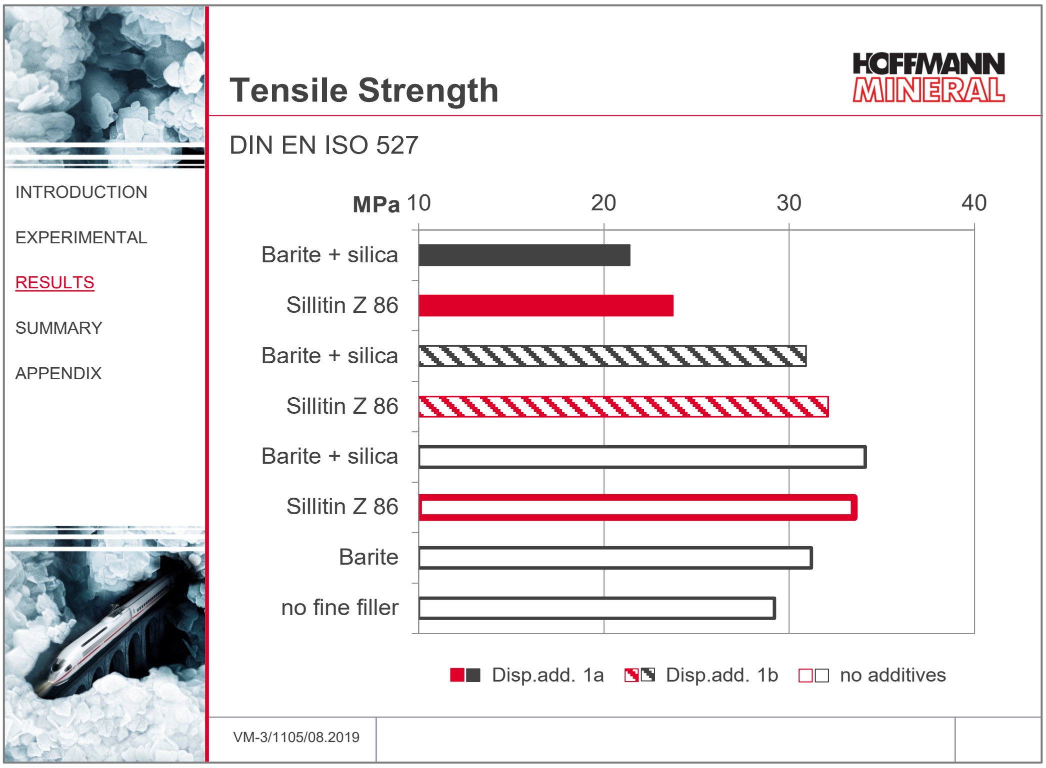
3.3 Mechanical properties

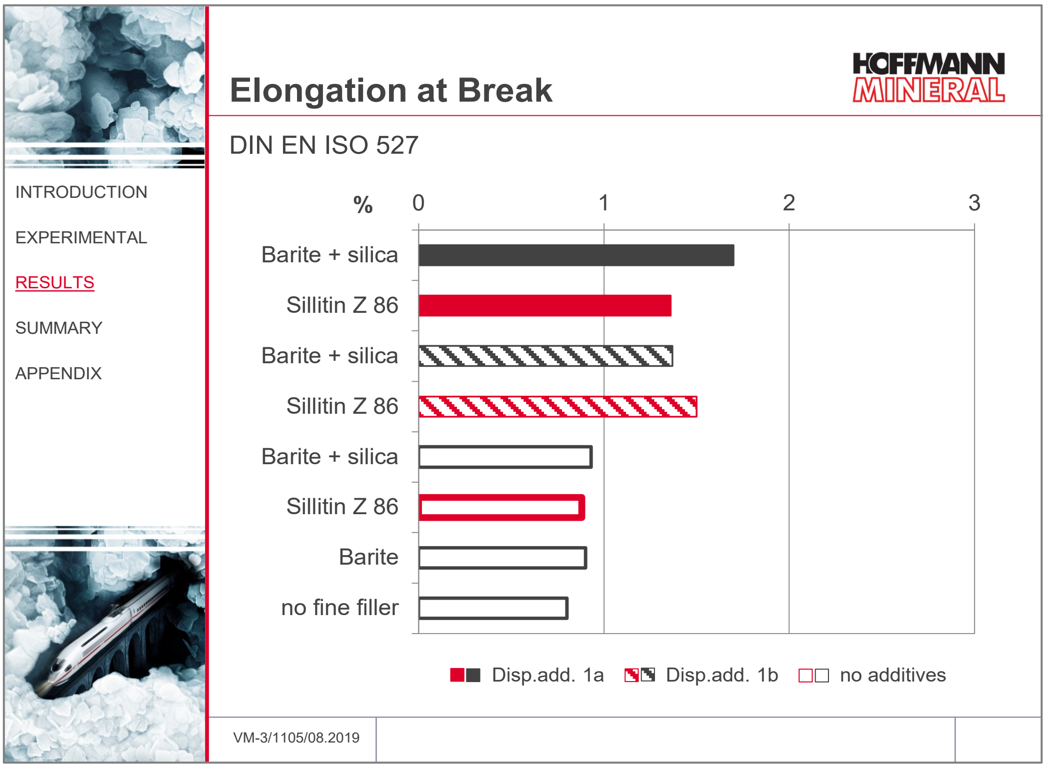
The required samples were prepared in appropriate casting molds, and were tested after complete cure (14 days at standard conditions, i.e. 23 °C and 50 % relative humidity).

Tensile test (DIN EN ISO 527)

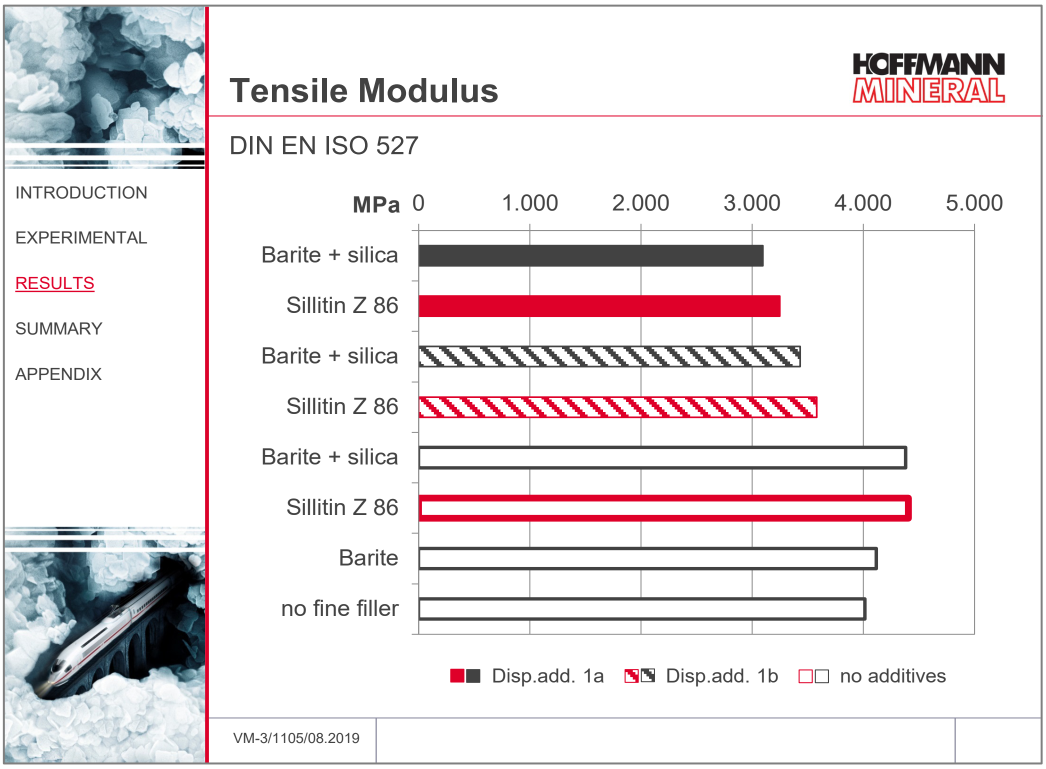
When working with the same additives, there is no significant difference between Neuburg Siliceous Earth and the barite compounds. Dispersion additive 1b or no additives at all will give markedly higher tensile strength compared with the base formulation.



Elongations at break come out at a similar level for Sillitin Z 86 and barite.

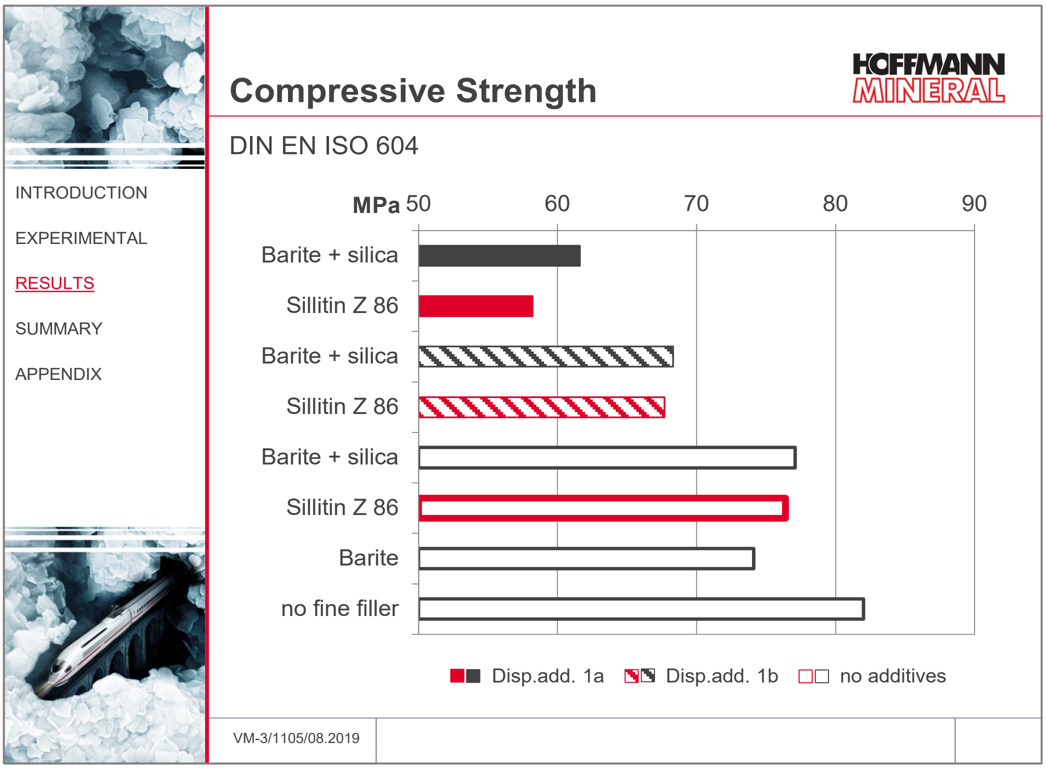


The tensile modulus too indicates a strong dependence on the additives used, while the fine filler containing compounds without additive exhibit the highest stiffness levels.



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.



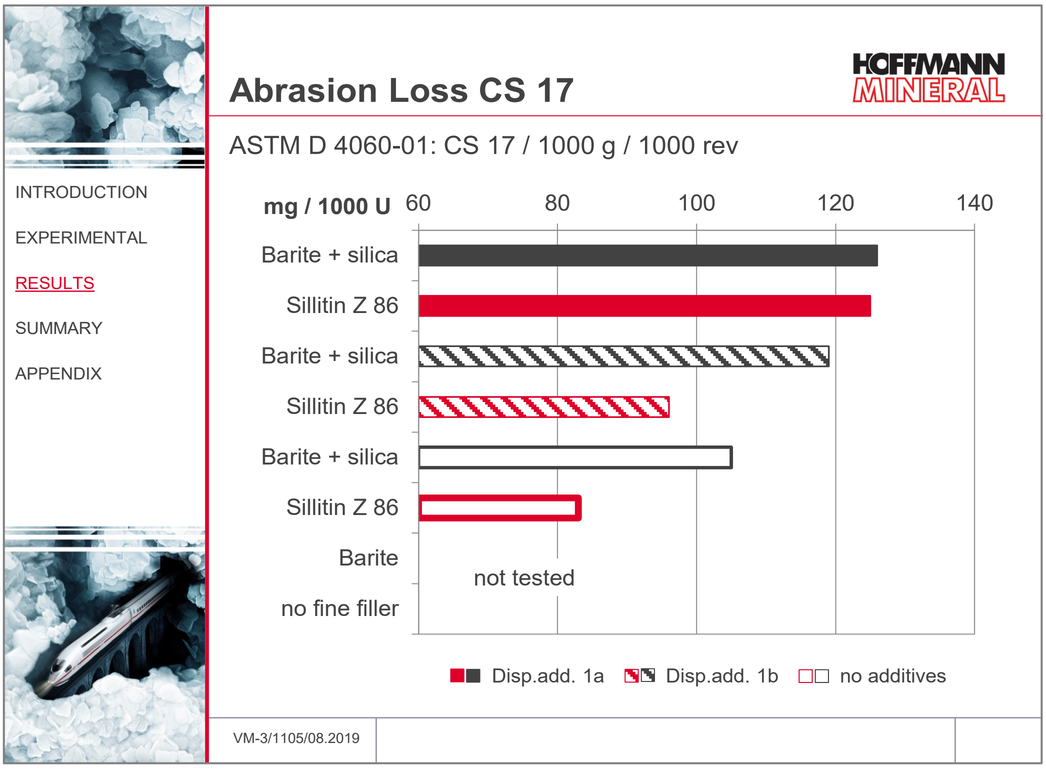
Hardness Shore D (DIN EN ISO 868)

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

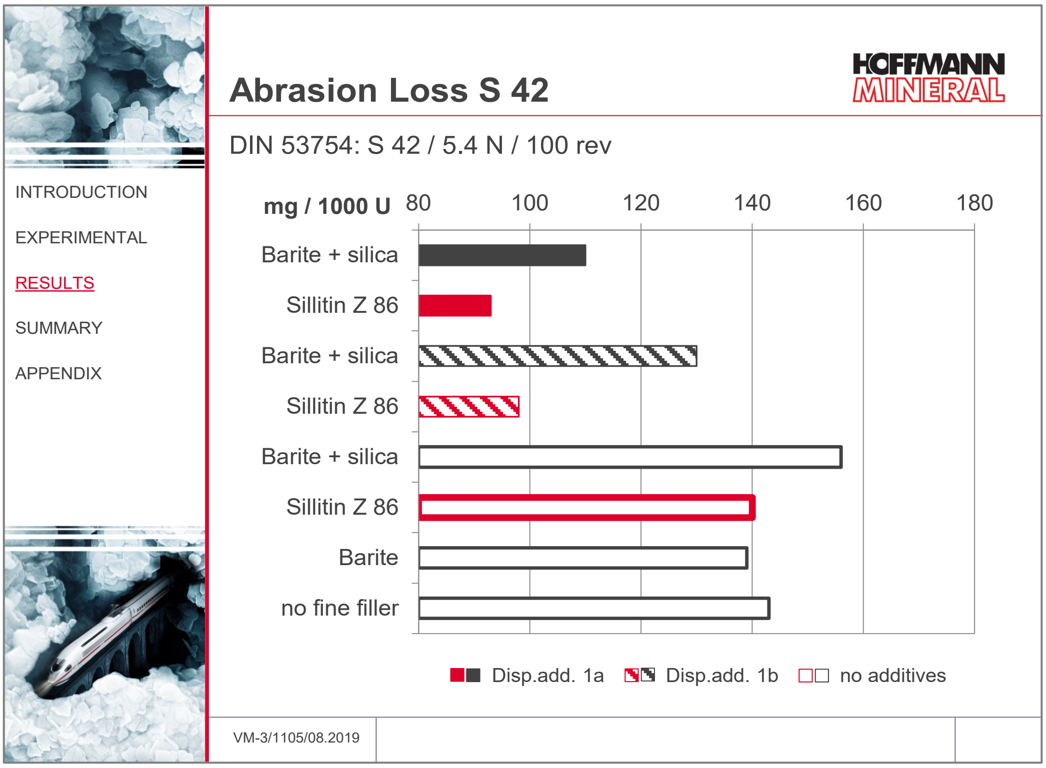
Abrasion loss according to DIN 53754 resp. ASTM 4060-01

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 compound formulated with Neuburg Siliceous Earth.

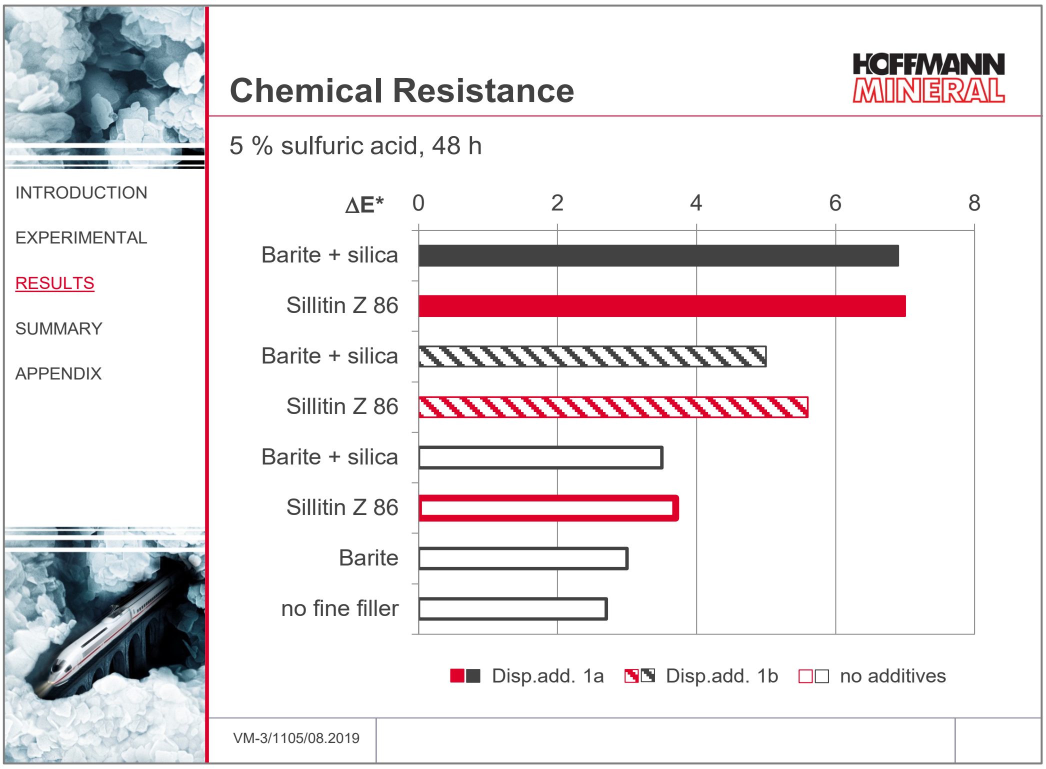


Against coarse abrasives (S 42), wear resistance falls off when additives are left out.



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 cross-over 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.

The improved processing properties are also maintained in sand extended formulations.

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

Appendix: viscosity