Calcined Neuburg Siliceous Earth as an anti-blocking agent in PET films

Author: Petra Zehnder
Hubert Oggermüller

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HOFFMANN MINERAL GmbH • Muenchener Strasse 75 • D-86633 Neuburg (Donau) • Phone +49 (0) 8431) 53-0
www.hoffmann-mineral.com • e-mail: info@hoffmann-mineral.com

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1 Introduction

In the manufacture of PET films, the addition of an anti-blocking additive is inevitably required in order to avoid the sticking together (blocking) of film layers at subsequent processing steps. The anti-blocking agent in addition should influence the optical properties of the films as little as possible, and give rise to a marked decrease of the coefficient of friction.

For optically demanding films, often synthetic silicas are used as anti-blocking additives. They mostly offer good optical properties, but in view of their high surface area they frequently affect the performance of other additives such as stabilizers or lubricants.

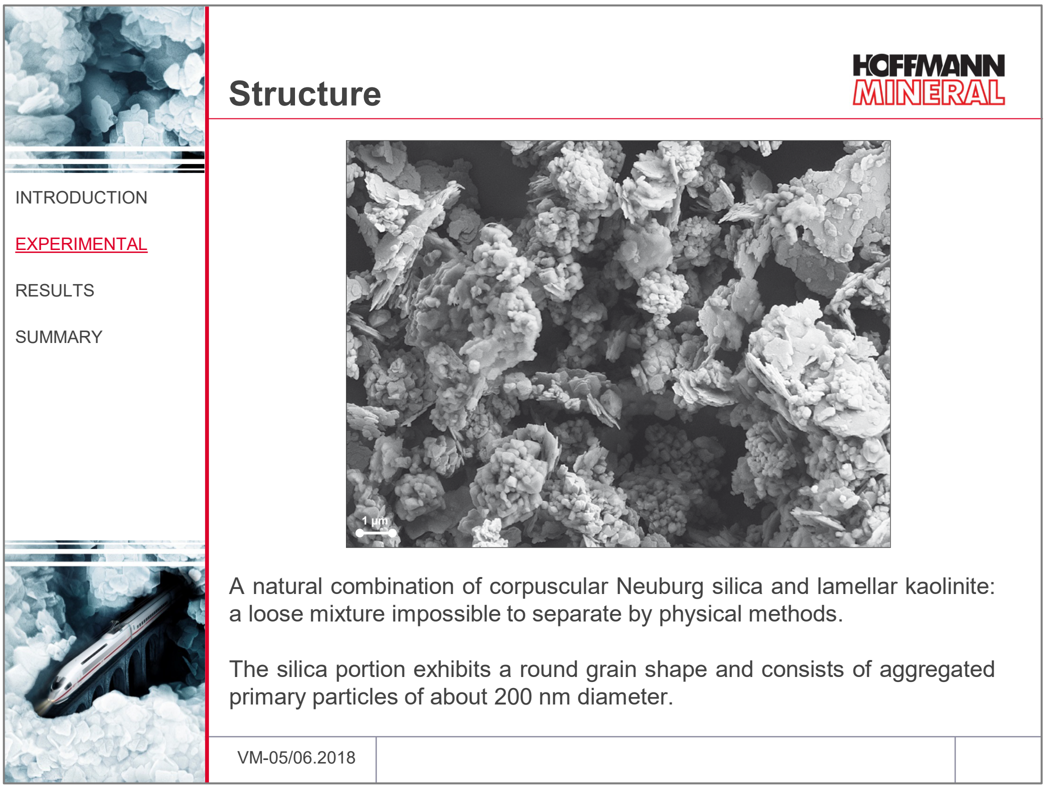
Calcined Neuburg Siliceous Earth, with natural silica as the major constituent, offers itself, based on its mineralogical composition and morphology, for use as an anti-blocking additive.

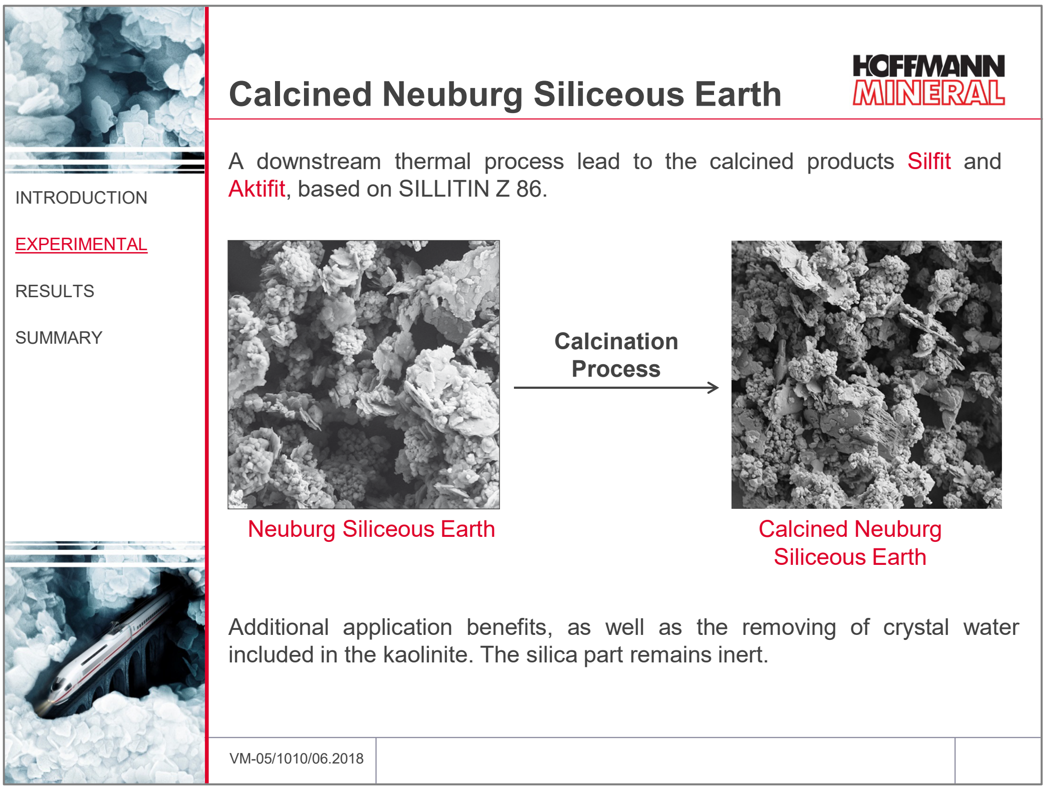
The present study has the objective to demonstrate the performance of calcined Neuburg Siliceous Earth as anti-blocking additive in PET films in comparison with traditional synthetic silicas.

2 Experimental

2.1 Neuburg Siliceous Earth

The special morphological composition of Neuburg Siliceous Earth, which represents a class of minerals on its own, in the following is illustrated by a SEM photograph.



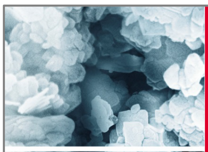


2.2 Mineral additives

As competitive additives, three synthetic silicas recommended for use with PET films were evaluated: a fumed grade, a high porosity precipitated silica and a further precipitated grade.

From the product portfolio of Neuburg Siliceous Earth, the calcined grade Silfit Z 91 was selected, because its morphology and mineralogical composition with natural silica as major constituent suggest a successful use as anti-blocking additive. In view of its low BET surface area, an interaction with other additives can practically be excluded.

The table summarizes the mineral additives evaluated and some of their typical properties.



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SUMMARY

Mineral Additives Characteristics

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	Particle size d_{50} [µm]	Specific surface area BET [m²/g]
Fumed silica	0.04 *	200
Precipitated silica 1 (silicagel type)	3.2	500
Precipitated silica 2	5	---
Silfit Z 91	2	7.5

* primary particle size
characteristics according to manufacturer

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2.3 Compounding and preparation of mono films

At first, masterbatches were prepared from a standard PET bottle grade with an intrinsic viscosity (IV) of 0.82 and the individual mineral additives. Silfit Z 91, as intended, could be incorporated into a masterbatch at 10 %, the synthetic silicas did only allow lower concentrations of 5 to 8 %.

For the subsequent processing to the final compound Invista 4027 was used, equally a standard PET grade with IV 0.61. The dosing of the masterbatch for each mineral additive was adjusted in a way to arrive at a concentration of 500 ppm (0.05 %) resp. 1000 ppm (0.1 %) in the final films.

Flat pre-films with a thickness of about 150 µm were extruded on a twin screw extruder ZSK 25 at 265 °C. Sections 85 x 85 mm were cut out and stretched biaxially following the simultaneous method (at the same time in longitudinal and transverse direction relative to the film extrusion). At an oven temperature of 90 °C and after 50 s preheating time the films were stretched with a rate of 100 %/s up to a stretch ratio of 3.5 x 3.5. There was no subsequent heat setting. The resulting final film had a thickness of about 15 µm.

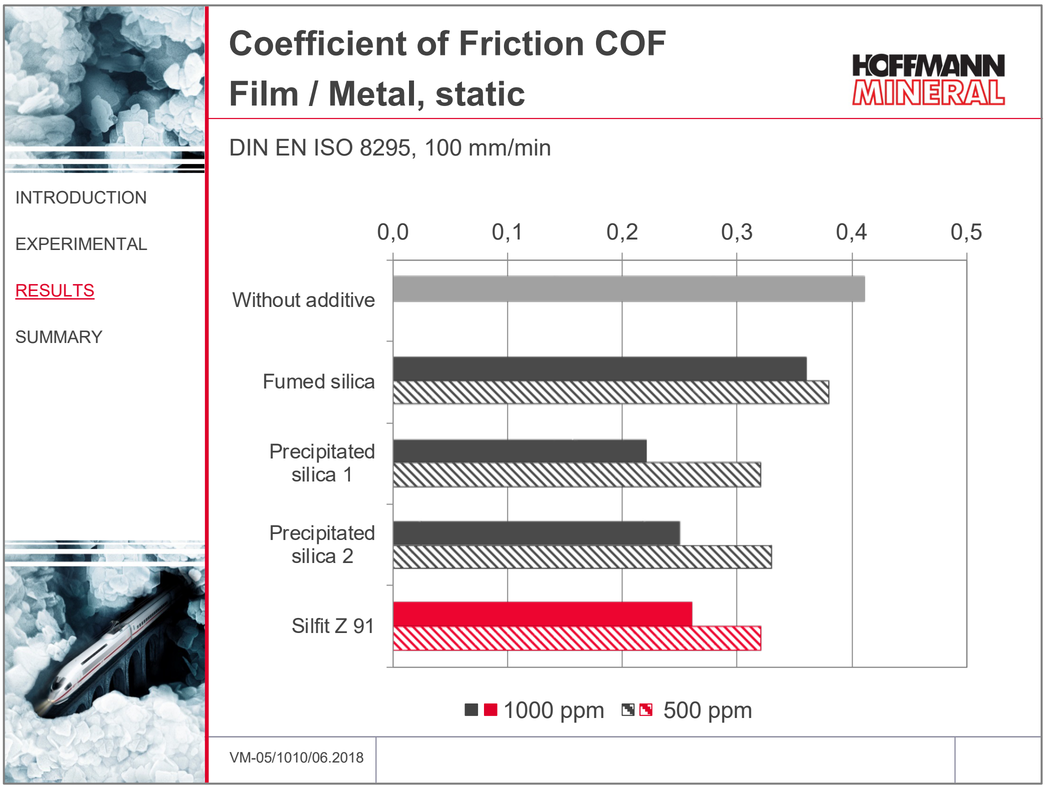
3 Results

3.1 Sliding behavior

The assessment of the sliding behavior was done via the coefficient of friction (COF). In the test according to DIN EN ISO 8295, a film section will be precisely fastened onto a slide-carriage (weight 200 g, base area 40 cm²). The slide is set without force onto the substrate in question. The test will be started after a waiting time of 15 s, then the slide will be drawn across the substrate with a speed of 100 mm/min. The test takes 10 s, whereby the first 2 s indicate the static friction (adhesion, static COF), and the remaining 8 s allow to calculate the sliding friction (dynamic COF). The lower the figures, the better the film slides over the substrate tested.

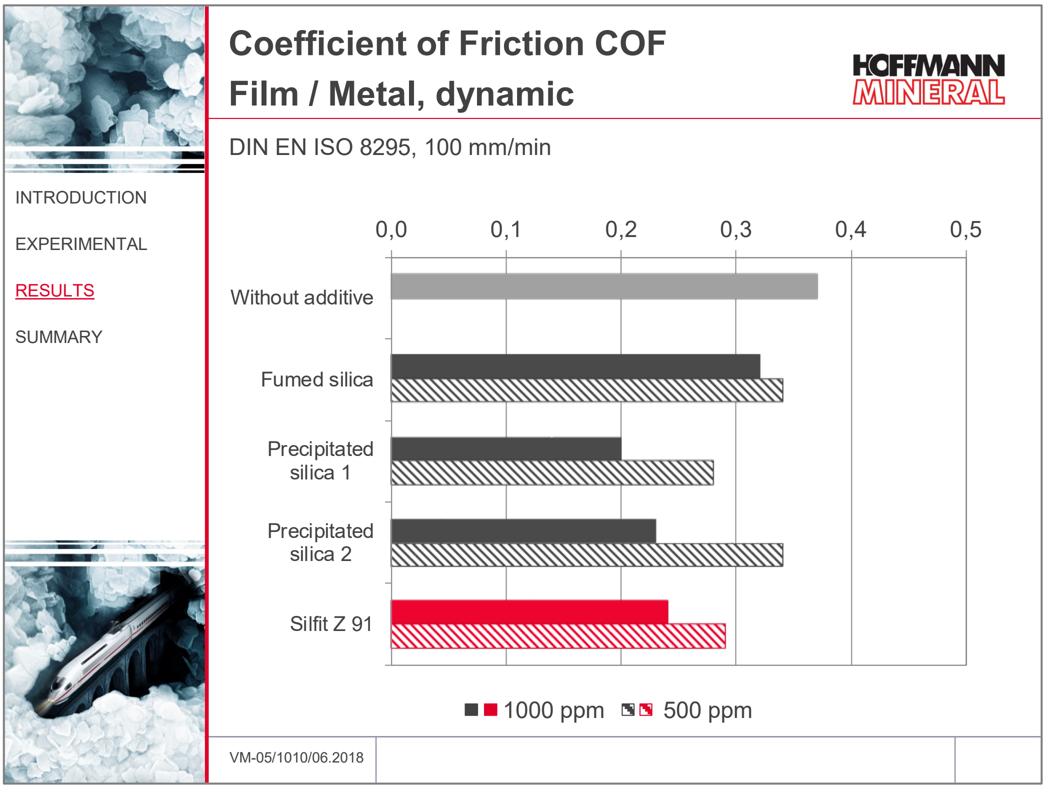
3.1.1 Coefficient of Friction Film to Metal

The coefficient of friction between film and metal indicates how well the film can be processed on fast running packaging machines.



The addition of a mineral additive in the case of Silfit Z 91 and the two precipitated silicas leads to a concentration dependent, but marked reduction of the coefficient of friction between film and metal. Silfit Z 91 comes up with similar sliding properties as the precipitated silicas.

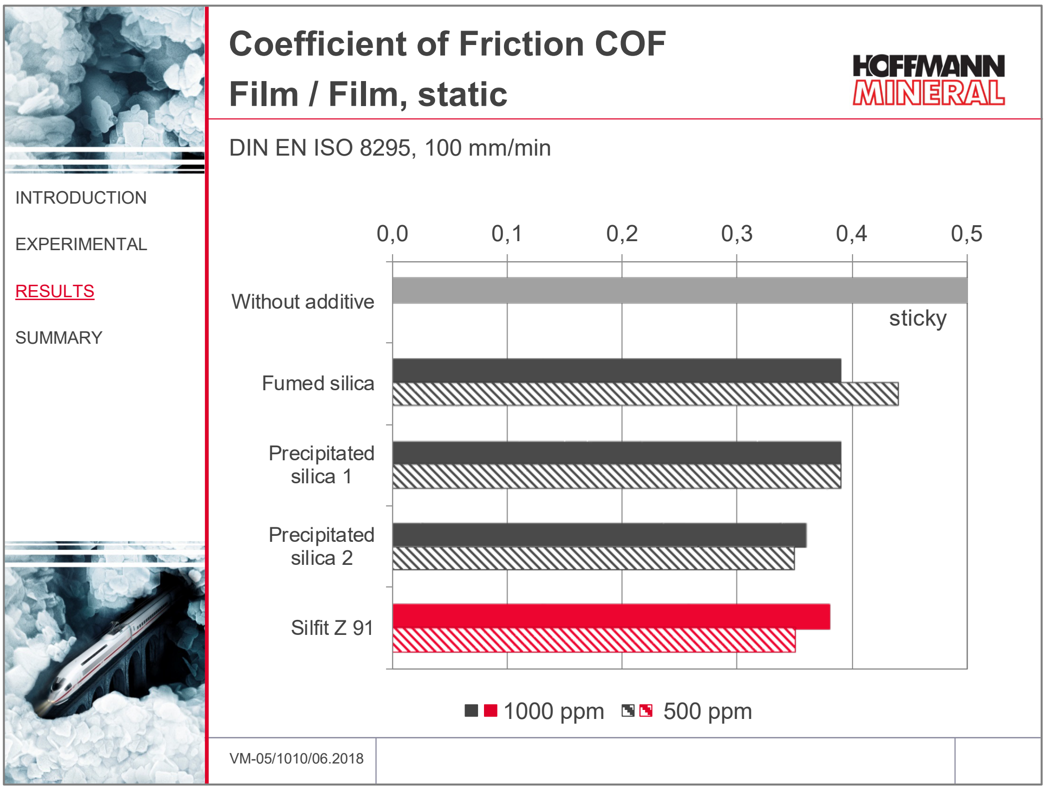
By contrast, the fumed silica shows clearly less efficiency. Even the higher loading of 1000 ppm affects the coefficient of friction film to metal only little.



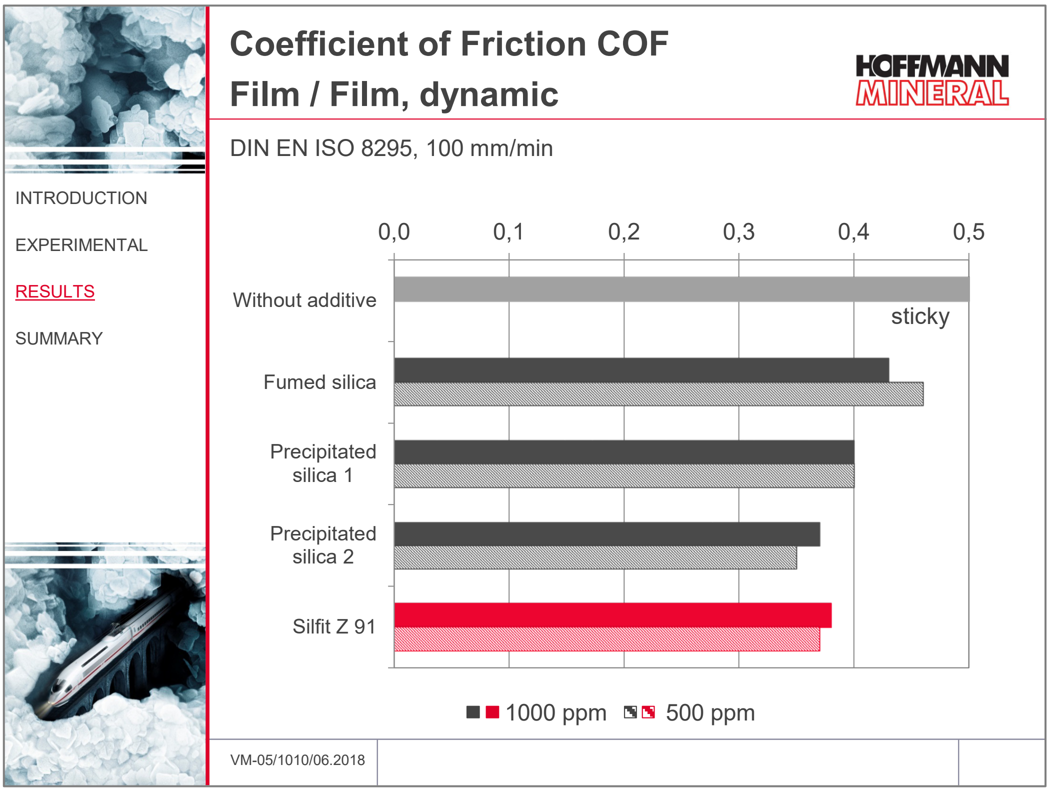
Such results are also observed for the dynamic COF between film and metal.

3.1.2 Coefficient of Friction Film to Film

The coefficient of friction film to film makes it possible to assess the behavior of films when processing film reels. The lower the COF, the less the individual film layers will tend to stick together.



It proved practically impossible to determine the coefficient of friction film to film of the samples without mineral additive. The film sections tended too strongly to stick to each other (blocking). The addition of a mineral additive gives rise to a marked decrease of the COF film to film, but a dependence of the concentration could not be established. Silfit Z 91 at least comes out at the same level as the substantially more costly silicas.



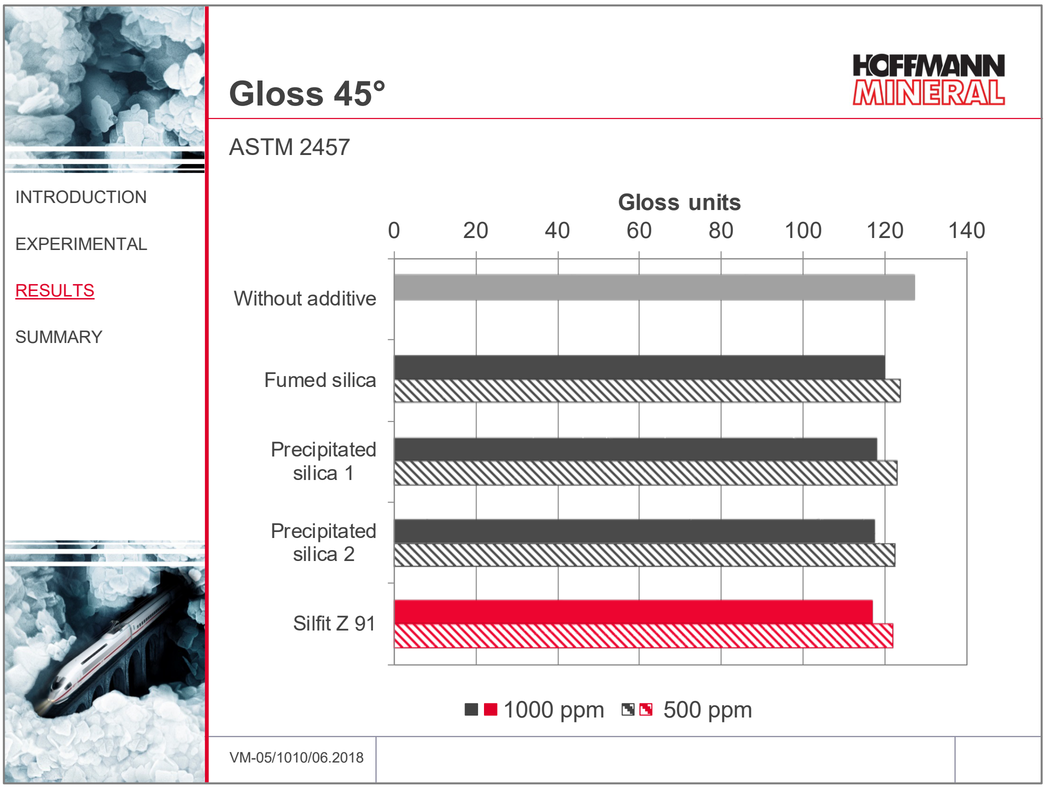
Under dynamic conditions the same situation is found as with the static COF film to film.

3.2 Optical properties

When the films are used for packaging purposes, frequently good optical properties are desired, such as high gloss and transparency along with as low a turbidity as possible.

3.2.1 Gloss 45°

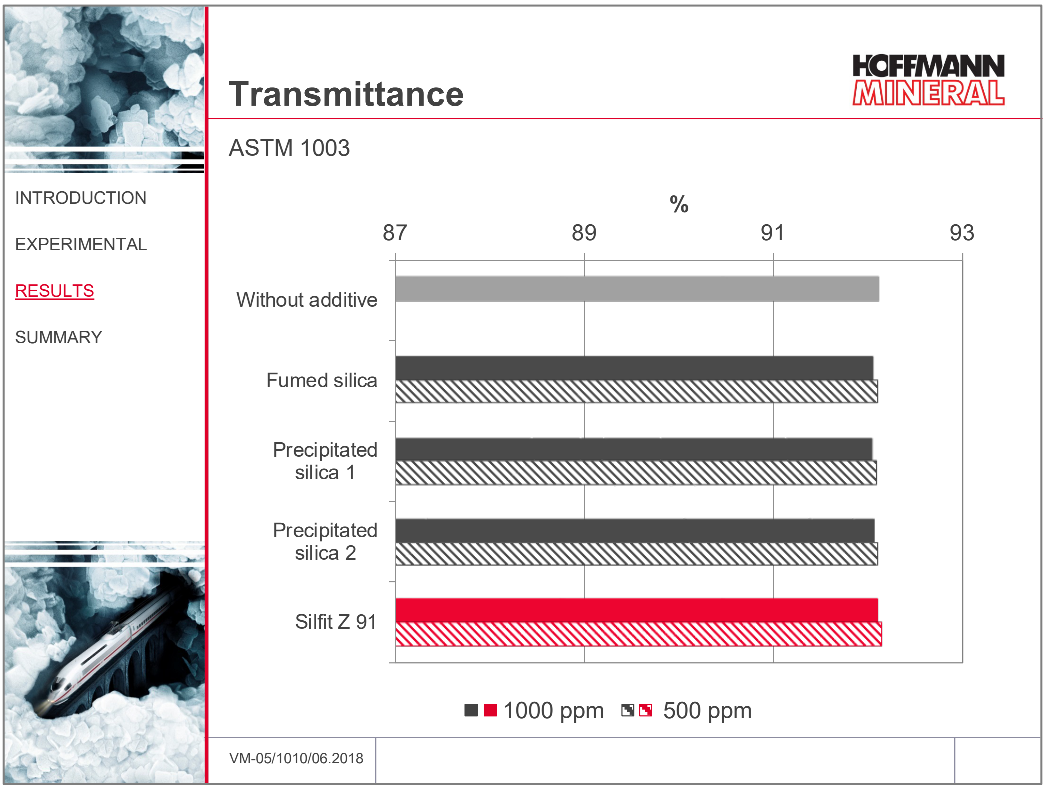
The gloss of the films was determined under a light entry angle of 45°.



The use of mineral additives, depending on the dosage, gives rise to minor decreased gloss. Between the individual additives, almost no differences can be observed.

3.2.2 Transmittance

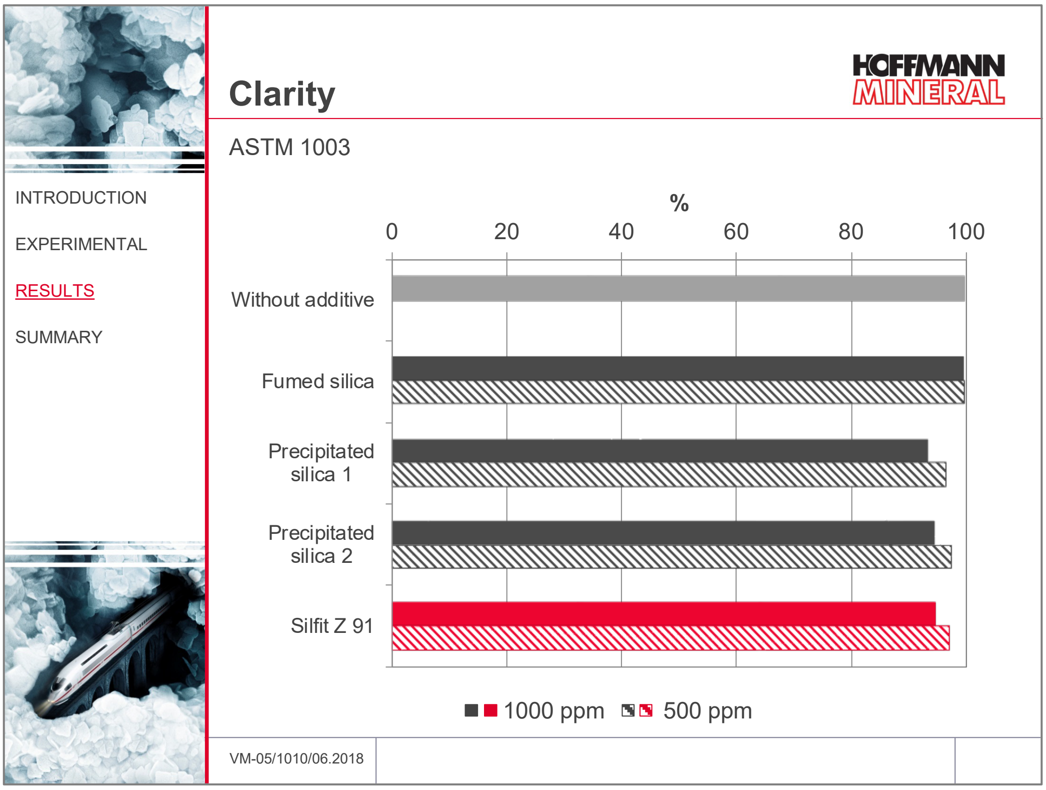
Transmittance is the ratio of transmitted to incoming light. It can be reduced by absorption and reflection. Films with a transmission of about 90 % are already considered as crystal-clear.



No significant influences can be detected, neither from the additive used nor from the concentration. The figures obtained are on level with the film without mineral additive.

3.2.3 Clarity

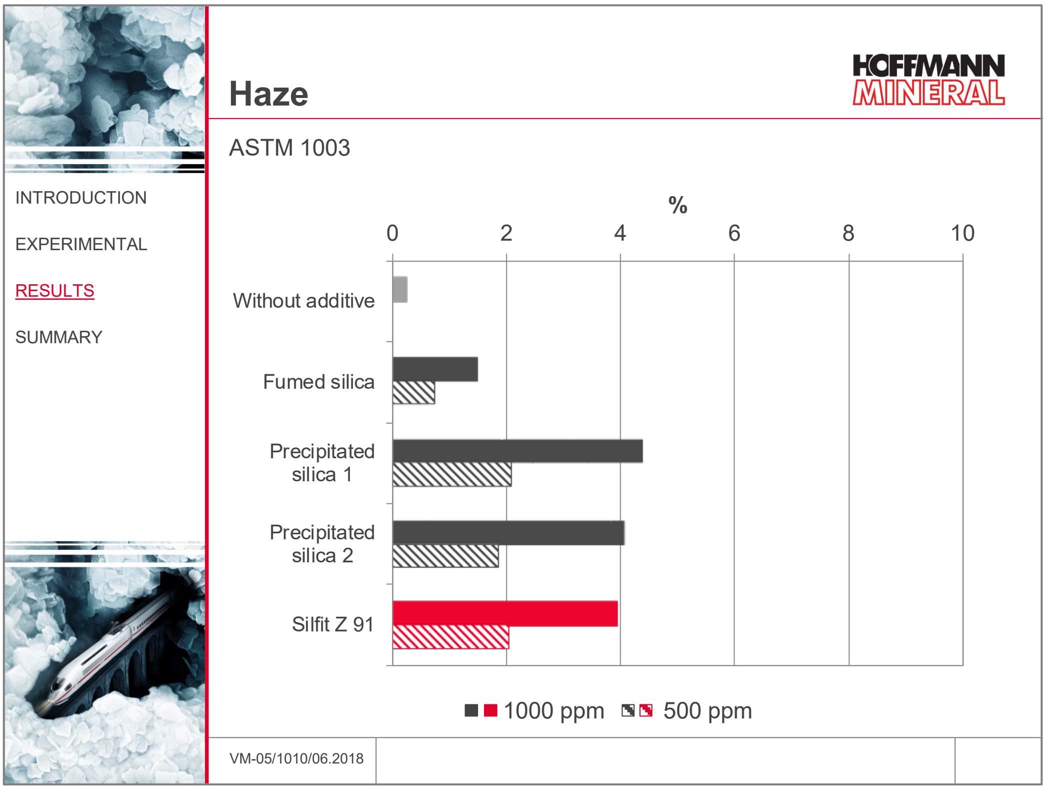
The transmitted quantity of light is divided into a directional and a diffuse portion. For the assessment of the clarity (image sharpness) the diffuse portion (scattered light) is examined in an angle range <2.5° (small angle scattering). This scattered light can distort contours which then appear less sharp. The higher the result, the sharper is the image when viewed through the film.



Different from the fumed silica, the two precipitated silicas as well as Silfit Z 91 give rise to a lesser adverse effect with respect to the clarity.

3.2.4 Haze

The turbidity (haze) of the film can be assessed via the scattered amount of light in the angle range >2.5° (large angle scattering). The higher the haze value, the more milky dull the film will appear, and the lower will come out contrast, transparency and gloss.



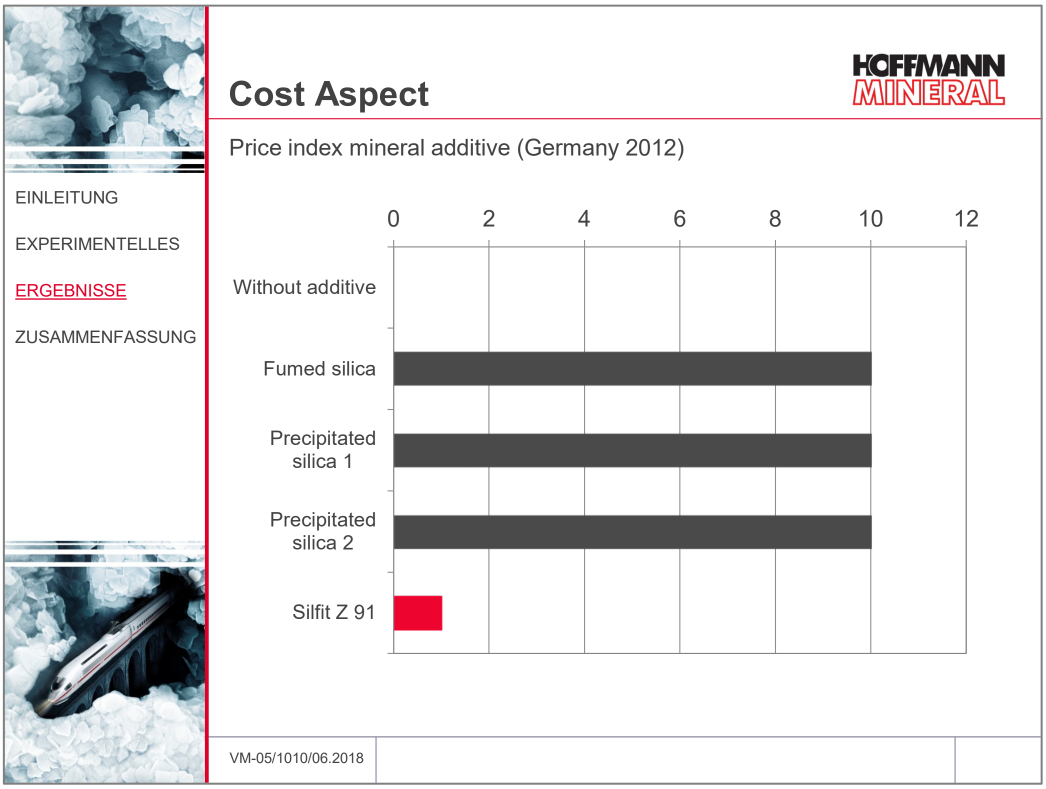
Similar to gloss, the haze of the film too will be influenced by mineral additives, and again there is a dependence on the concentration.

The fumed silica used here marks the best result with only 0.7 resp. 1.5 %. This has, however, to be put into perspective by the comparably poor results for the coefficients of friction.

The precipitated silicas give haze results about twice as high, but they lead to better coefficients of friction than the fumed grade.
Silfit Z 91 remains fully comparable with the precipitated silicas.

4 Cost aspect

For the price comparison, the straight price of the mineral additive will be set in relation with Silfit Z 91 (= 1).



The price of Silfit Z 91 is only about one-tenth of what has to be spent for the traditionally used silicas.

5 Summary

Silfit Z 91 in comparison with anti-blocking additives based on synthetic silicas is characterized by a performance profile similar to precipitated silicas. Along with a low coefficient of friction, good optical properties will be obtained.

The high bulk density stands for a low tendency towards dust formation. As a mineral additive, Silfit Z 91 is easily dispersed. Interactions with other additives can practically be excluded in view of the low BET surface area.

Compared with the synthetic silicas, which often are used for demanding films, there exists a significant advantage in cost.

Overall already low loadings of Silfit Z 91 give rise to good anti-blocking efficiency, along with just a slight impairment concerning the optical properties. In summary, Silfit Z 91, therefore, can be considered as very well suited for use as a cost effective anti-blocking agent for PET films.

Supplement:

Aktifit VM, a calcined Neuburg Siliceous Earth grade surface treated with a special vinyl functional group, is expected to achieve the same properties in the film. An additional advantage of Aktifit VM, however, is the very low moisture content of the filler without moisture absorption in humid climatic conditions.