Unternehmensgruppe Hoffmann

Neuburg Siliceous Earth as anti-blocking agent in thin LLDPE-films

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1 Introduction

For the production of LLDPE films, anti-blocking agents are used to improve sliding friction. The optical properties of the film should be affected as little as possible.

The disadvantage of organic slip additives based on erucamic acid (ESA) or oleic acid is that they migrate over time, which leads to a change in sliding friction. As a consequence, their dosage depends on the film thickness, the thinner the film, the higher the concentration must be chosen. In addition, the solubility of slip additives in the polymer increases with rising temperature, which also changes the processing properties of the film.

Mineral additives such as diatomaceous earth, talc or synthetic silica increase the surface roughness of the film and thus improve the sliding friction. However, a high dosage often deteriorates the optical properties of the film. Synthetic silicas usually achieve good optical properties, but often reduce the effect of other additives such as stabilizers, lubricants etc. due to their high specific surface area.

Therefore, organic slip additives are usually combined with mineral anti-blocking additives.

Neuburg Siliceous Earth, with natural silica as the major constituent, lends itself, based on its mineralogical composition and morphology, to the application as anti-blocking additive.

In the present study, the performance of Neuburg Siliceous Earth as a mineral anti-blocking additive in LLDPE films is shown in comparison with commercially available competitive minerals.

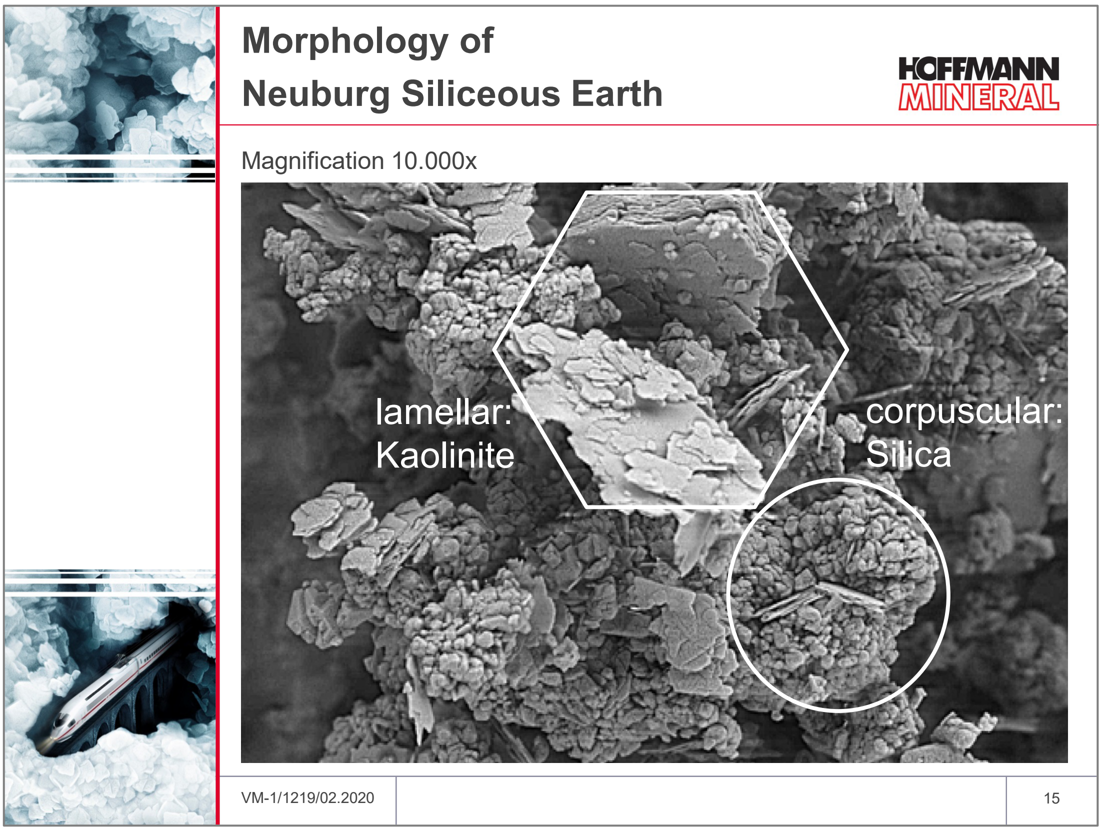
Slip additives were deliberately omitted in order to clearly show the effects of the various mineral additives.

2 Experimental

2.1 Neuburg Siliceous Earth

Classic Neuburg Siliceous Earth is a natural combination of corpuscular Neuburg Silica and lamellar kaolinite: a loose mixture impossible to separate by physical methods. The silica portion exhibits a round grain shape and consists of aggregated primary particles of about 200 nm diameter.

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.



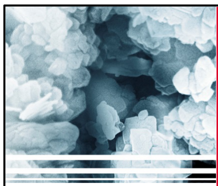
The basis for the calcined Neuburg Siliceous Earth is the standard product Sillitin Z 86. In a thermal process, the water of crystallization in the kaolinite portion is removed and new, largely amorphous mineral phases are formed.

The resulting product Silfit Z 91 is characterized by high brightness and color neutrality.

The Aktifit grades are special products where Silfit Z 91 has been surface treated with functional additives.

2.2 Mineral additives

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



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Mineral Additives Characteristics

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	Particle size d_{50} [µm]	Particle size d_{97} [µm]	Oil absorption [g/100g]	Specific surface area BET [m²/g]	Funcionali-sation
Diatomaceous earth	6.8	28	130	2,9	no
Precipitated silica	4.8	9	160	420	no
Talc	3.4	13	45	14	no
Sillitin V 88	4.0	18	45	8	no
Sillitin Z 89 puriss	1.9	9	55	11	no
Silfit Z 91	2.0	10	65	10	no
Aktifit PF 111	2.0	10	60	9	alkyl hydrophobic

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As comparative additives, three mineral additives common for LLDPE films were used: diatomaceous earth, a precipitated silica (silica gel type) and a special soft talc type.

From the product range of Neuburg Siliceous Earth, which due to its morphology and mineralogical composition with natural silica as the main component is suitable for use as an anti-blocking additive, a total of four grades were selected.

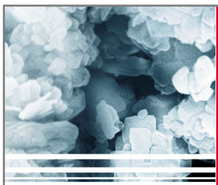
Sillitin V 88 and Sillitin Z 89 puriss are two classic standard products, with Sillitin V 88 being a coarser grade and Sillitin Z 89 puriss a finer grade with particularly good dispersion properties. Silfit Z 91 is the particularly color-neutral calcined base product with similar particle size distribution and dispersion behavior as Sillitin Z 89 puriss.

Aktifit PF 111 is an activated Silfit Z 91, which has been hydrophobized by surface treatment with an alkyl functional group.

Due to the low BET surface area of Neuburg Siliceous Earth grades, interaction with other additives can be practically excluded.

2.3 Compounding and preparation of blown films

An LLDPE grade suitable for blown film applications, Sabic 118 N with a volume flow rate MVR of 1.0 g/10 min at 190 °C, was selected for the production of the films. Final compounds with 3000 and 10000 ppm (resp. 0.3 and 1.0 %) mineral additive were prepared from the masterbatches containing 5 % mineral additive. Further processing into films with a thickness of 20 µm was carried out on a blown film line under the conditions given in the table.



Compounding
Film Preparation

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Masterbatch	95 % LLDPE Sabic 118 N (MVR 1 g/10 min) 5 % mineral additive
Final compound	LLDPE Sabic 118 N + Masterbatch
Content of mineral additive in the film	3000 ppm resp. 0.3 % 10000 ppm resp. 1.0 %
Film blowing	Melt temperature 182-184 °C Nozzle Ø 60 mm, annular gap 2 mm Blow-up Ratio (BUR) 2.5 Haul-off speed 10.5-11 m/min Film thickness 20 µm

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3 Results

3.1 Sliding behavior

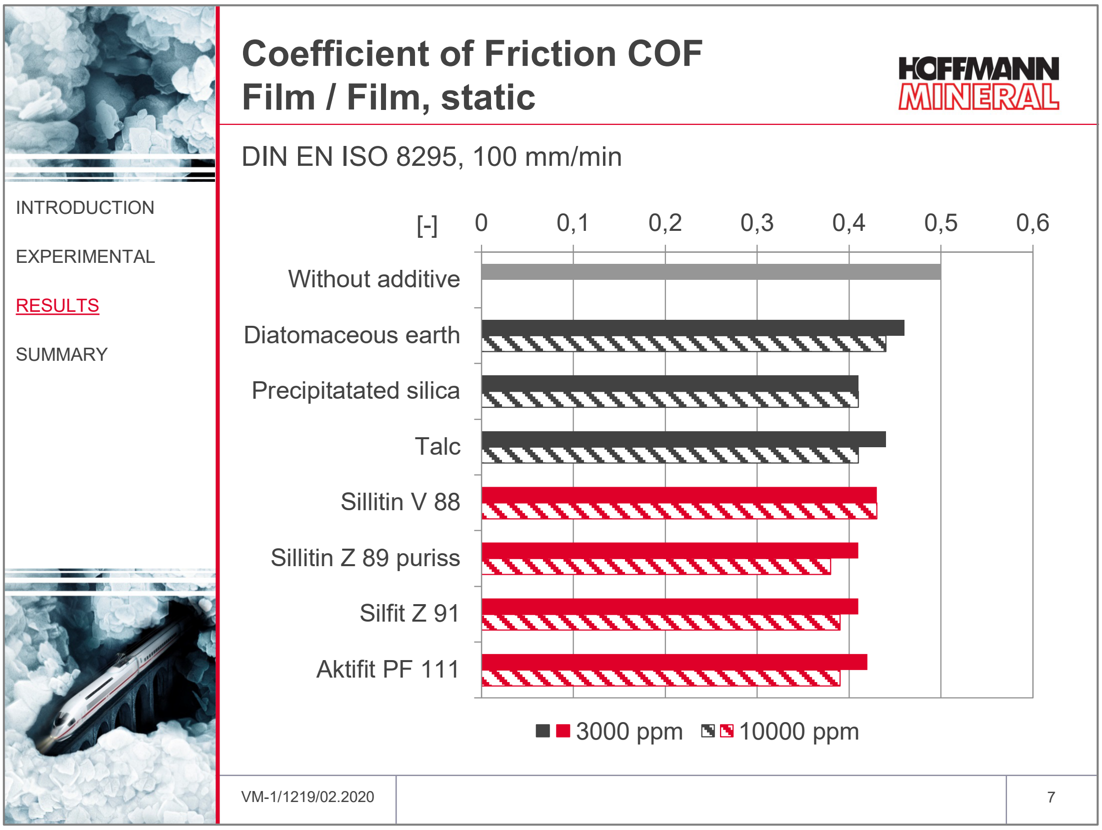
The sliding behavior was assessed by means of the coefficient of friction (COF).

For testing according to DIN EN ISO 8295, a section of film is precisely attached to a slide-carriage (weight 200 g, base area 40 cm²). The slide is placed on the corresponding substrate (foil or metal) without force. After a waiting time of 15 s the measurement is started and the slide is drawn over the substrate at a speed of 100 mm/min. The measurement 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 smaller the value, the easier the film glides over the selected substrate

3.1.1 Coefficient of friction film to film

The coefficient of friction film/film can be used to assess the behavior of films when processing film reels. The lower the COF, the less the individual film layers tend to stick together on the roll.

In each case, the inner sides of the film tube were measured in the machine direction of the film.



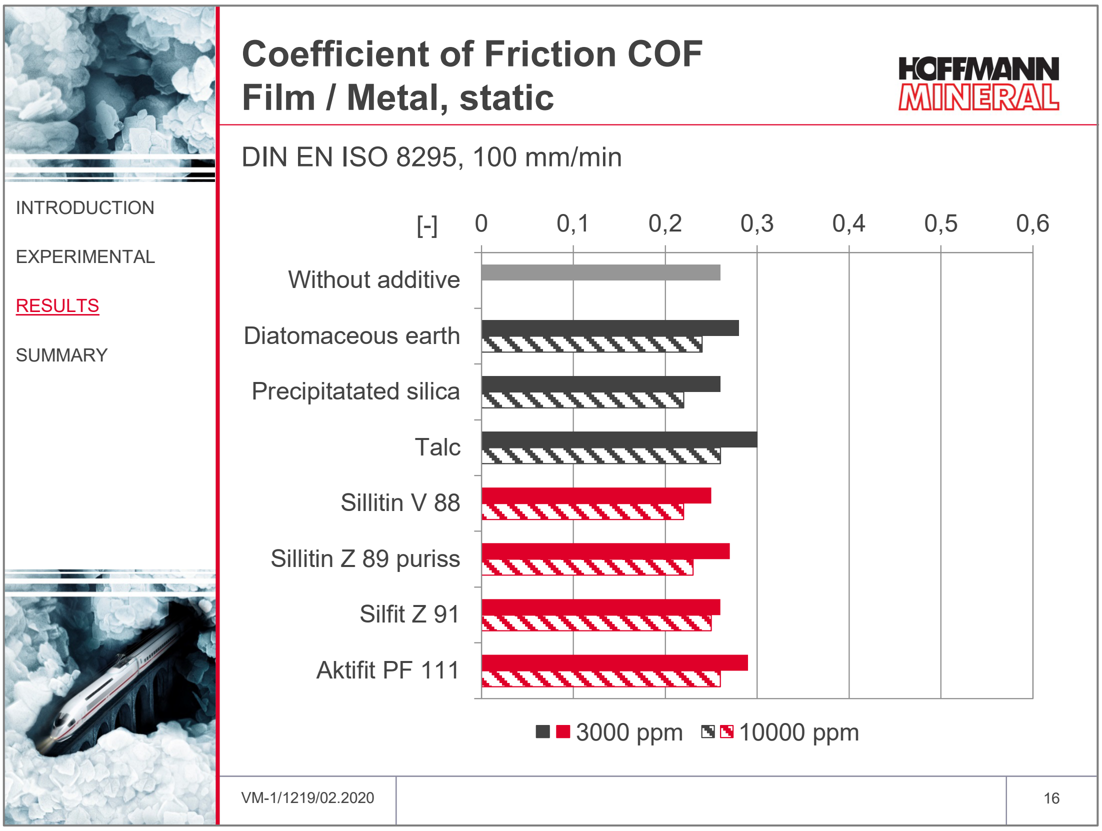
With regard to the coefficient of friction film/film, improved sliding properties can already be observed with the lower dosage of the mineral additive compared to the unfilled film. By increasing the dosage, this effect is even slightly enhanced. The finer grades of Neuburg Siliceous Earth, Sillitin Z 89 puriss, Silfit Z 91 and Aktifit PF 111, show the best results here. The coarser Sillitin V 88 is better suited for thicker films.

There is no significant difference between static and dynamic COF.

3.1.2 Coefficient of friction film to Metal

The coefficient of friction film/metal provides information on how the film can be processed on high-speed packaging machines.

Here too, the measurement was made on the inside of the film tube in the machine direction of the film.



In the low dosage of 3000 ppm, the sliding properties film/metal are hardly influenced by the addition of the mineral additive and remain at the level of the unfilled film.

Only the higher dosage of 10000 ppm causes a reduction of the friction coefficient film/metal. Here, Neuburg Siliceous Earth, especially the standard grades Sillitin V 88 and Sillitin Z 89 puriss, together with the precipitated silica, achieve the best results.

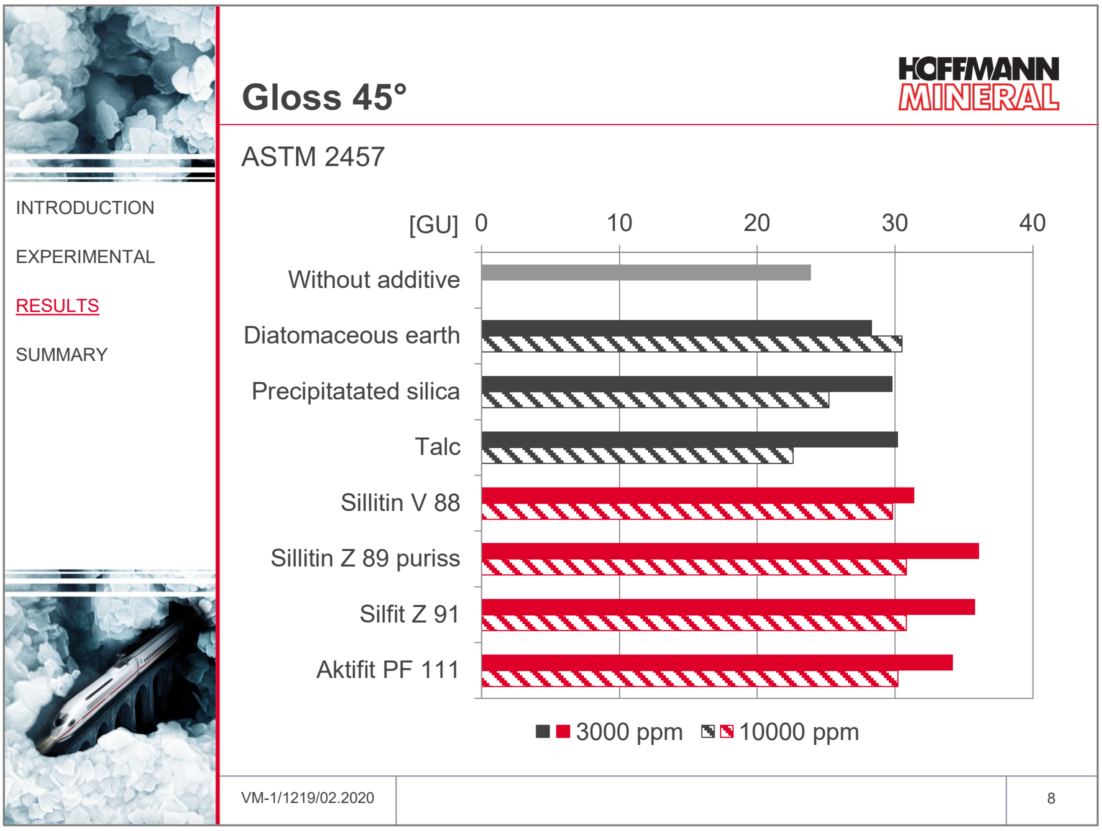
There is also no difference between static and dynamic COF film to metal.

3.2 Optical properties

When using the films in the packaging sector, good optical properties such as high gloss and transparency and the lowest possible haze are frequently desired.

3.2.1 Gloss 45°

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

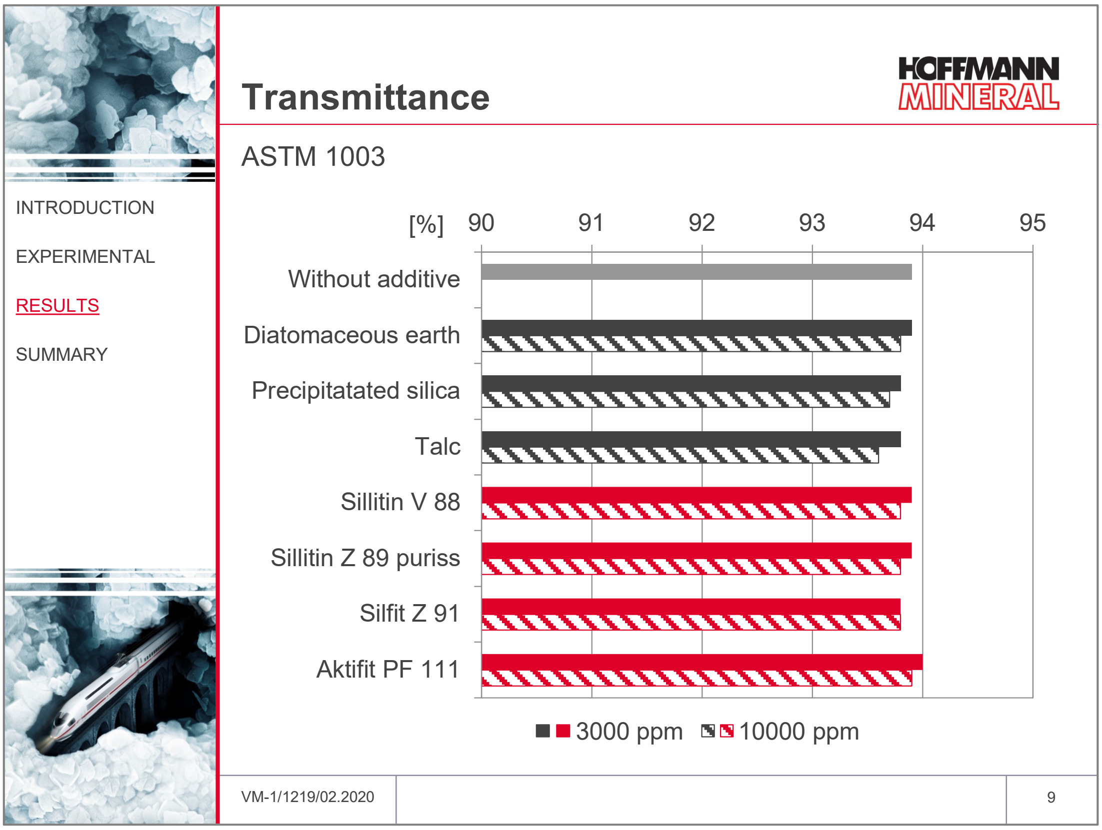


The use of 3000 ppm mineral additive noticeably increases the gloss of the film, with the fine grades of Neuburg Siliceous Earth achieving the highest values. Increasing the dosage to 10000 ppm reduces the gloss of talc and precipitated silica back to the level of the unfilled film. Diatomaceous earth and especially Neuburg Siliceous Earth in the higher dosage give films with higher gloss.

The reason for the higher gloss should be a more homogeneous surface with more directional light reflection, which in turn - with unchanged process parameters - could be due to a reduction in crystallinity or smaller spherulites.

3.2.2 Transmittance

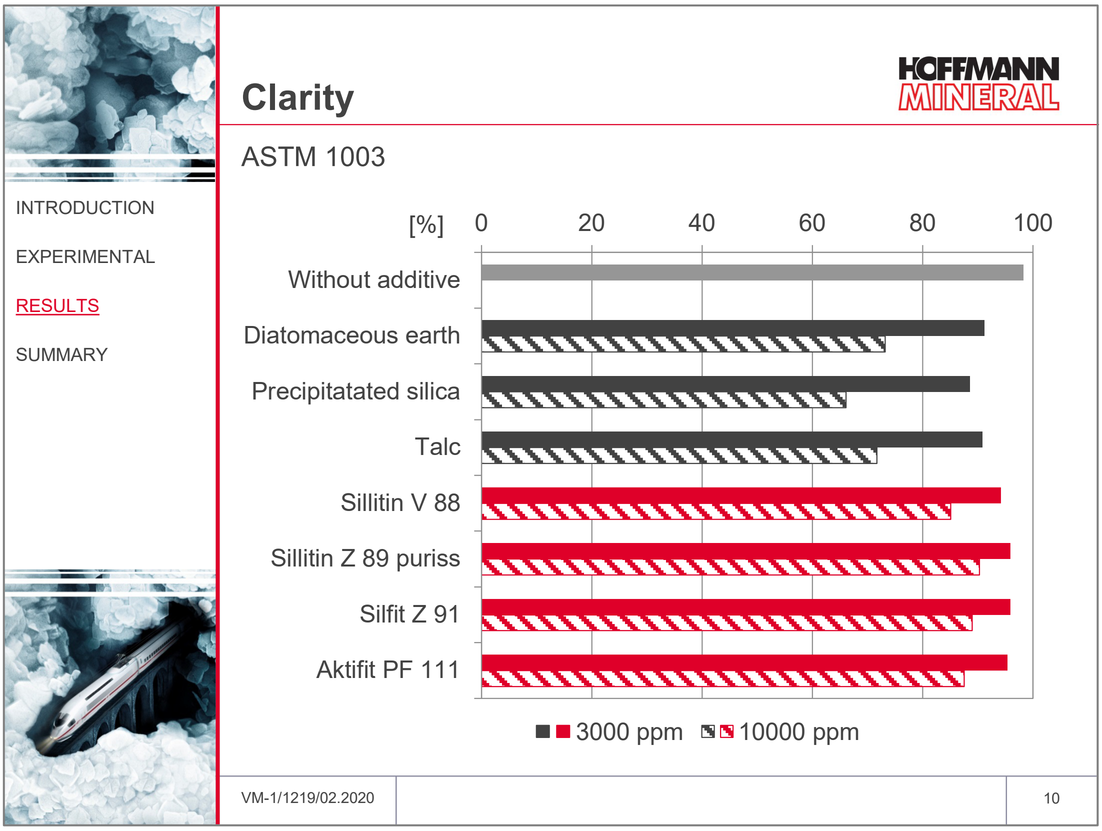
The transmittance (light transmission) is the ratio of transmitted light to incident light. It can be reduced by absorption and reflection. Films with approx. 90 % transmission are already considered crystal clear.



In the case of transmittance, there is a trend towards slightly lower values. In higher dosage, talcum reduces light transmission most significantly. Neuburg Siliceous Earth grades reach the highest values.

3.2.3 Clarity

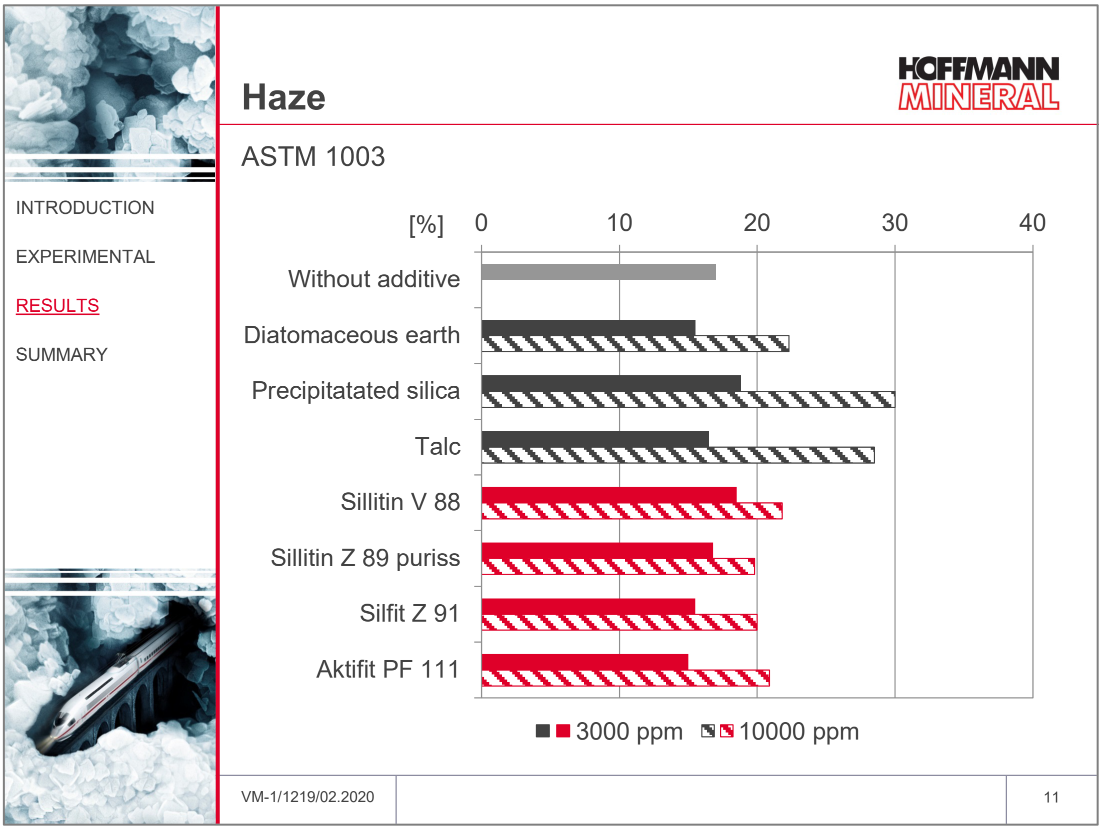
The transmitted light quantity is divided into a directional and a diffuse part. For the evaluation of the clarity (image sharpness) the diffuse part (scattered light) is considered in an angular range < 2.5° (small angle scattering). This scattered light distorts contours and makes them appear less sharp. The higher the value, the sharper the image when viewed through the film.



The addition of mineral additive results in a reduction of image sharpness for all materials, clearly visible in the higher dosage of 10000 ppm. With Neuburg Siliceous Earth, the image sharpness is significantly less affected than with the competing minerals, and in the low concentration the result of the film without additive is approached.

3.2.4 Haze

The turbidity (haze) of the film can be assessed by the amount of scattered light in an angular range > 2.5° (wide angle scattering). The higher the haze value, the milkier the film will appear and the lower will come out contrast, transparency and gloss.



Analogous to the reduced image sharpness, the cloudiness caused by mineral additives increases accordingly in the higher dosage.

Among competitors, talc and precipitated silica lead to a significantly higher turbidity than diatomaceous earth. Neuburg Siliceous Earth, especially the finer grades Sillitin Z 89 puriss, Silfit Z 91 and Aktifit PF 111, show by far the lowest haze of the film.

4 Summary

Neuburg Siliceous Earth is very well suited as mineral anti-blocking additive for LLDPE films.

While a low coefficient of friction is already achieved in low dosage, the good optical properties remain at a good level even in higher dosage. Due to the high bulk density there is only a low tendency towards dust formation. As a mineral additive, Neuburg Siliceous Earth is easily dispersible. Interactions with other additives (e.g. slip additives) can be largely excluded due to the low BET surface area. Compared with synthetic silicas, which are often used for optically demanding films, there is a significant cost advantage

Product recommendations:

Sillitin V 88	cost-effective standard grade, especially for films with higher thickness
Sillitin Z 89 puriss	better optical properties, higher gloss, high clarity and lowest haze
Silfit Z 91	same as Sillitin Z 89 puriss, additionally higher color neutrality
Aktifit PF 111	same Silfit Z 91, but hydrophobic, very low moisture content without increase in humid climatic conditions, minimized interaction with slip additives