Making Sure that Nothing Blocks

Calcined Neuburg Siliceous Earth as an Antiblock Additive in PET Films

By means of antiblock additives it is possible to manufacture films without adhesion and non-blocking. In practice, synthetic silicas are frequently used as antiblock additives for PET films. Recent investigations have shown that also calcined Neuburg siliceous earth is suitable – with numerous advantages.

Non-adhesive and non-blocking PET film with natural minerals as antiblock additive (figure: Fotolia)

When producing PET films (**Title figure**), the use of an antiblock additive is necessary to prevent the mutual adherence of film layers (blocking) during subsequent processing. Hereby, the antiblock additive must have a minimum effect on the film's optical properties, and should greatly reduce its coefficient of friction.

For optically demanding films, synthetic silicas are frequently used as antiblock additives. Although they mostly have good optical properties, their large specific surface area (BET) often reduces the influence of other additives such as stabilizers or lubricants.

Due to its mineralogical composition and morphology, calcined Neuburg siliceous earth with natural silica as main constituent (**Fig. 1**) is very well suited as an antiblock additive. This article describes the performance of calcined Neuburg siliceous earth as an antiblock additive in PET films, and compares it with traditional synthetic silicas.

Suitability Test

In order to investigate the suitability of calcined Neuburg siliceous earth as an antiblock additive in PET films, three synthetic silicas – which are recommended for PET films – were used: a pyrogenic type, a highly porous precipitated silica (silica gel type), and another precipitated silica.

Calcined Silfit Z 91 was selected from the range of Neuburg siliceous earth products, because its morphology and mineralogical composition with natural silica as main constituent makes it suitable for use as an antiblock additive. Due to the lower BET surface area (compared with synthetic silicas), an interaction with other additives can be practically excluded. **Table 1** gives an overview of the mineral additives used, and their most significant characteristics.

From Masterbatch to Film

To start with, masterbatches based on a standard PET bottle type with an intrinsic viscosity IV of 0.82 were created with the corresponding mineral additives. Hereby – as planned – Silfit Z 91 could be added to the masterbatch in an amount of 10 %.

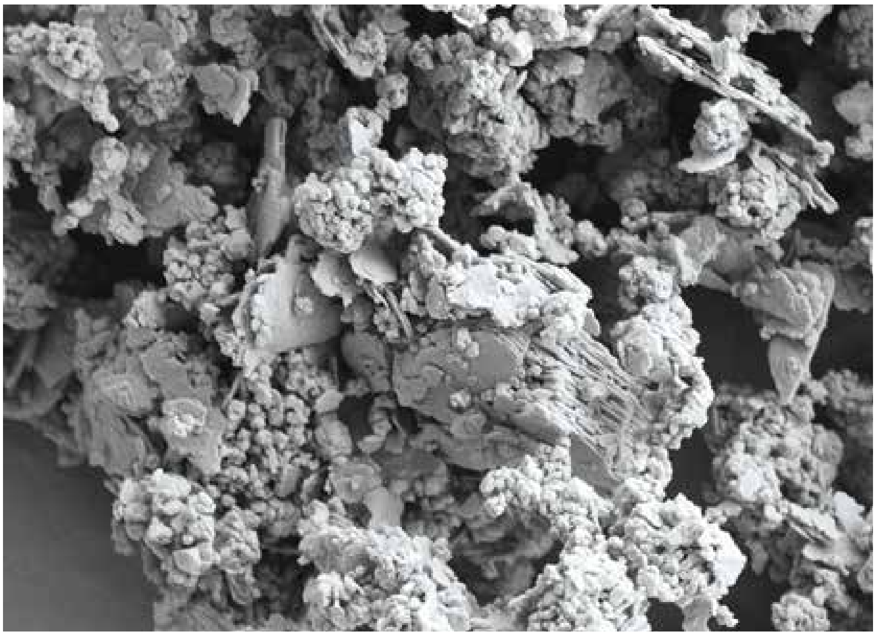

Fig. 1. REM image with 10,000-fold magnification of the structure of calcined Neuburg siliceous earth (figures: Hoffmann Mineral)

Mineral additives	Particle size d50 [µm]	Specific surface area BET [m^2/g]
Pyrogenic silica	0.04*	200
Precipitated silica 1 (silica gel type)	3.2	500
Precipitated silica 2	5	--
Silfit Z 91	2	7.5
* Primary particle size		

Table 1. Characteristics of mineral additives (manufacturer's data)

The synthetic silicas had proportions of only 5 to 8 %.

For further processing into a compound, the PET material Invista 4027 (manufacturer: Invista (Deutschland) GmbH in Hattersheim, Germany) was used. This is also a standard PET type with a viscosity of 0.61. Masterbatch dosing was adapted for every additive, so that their subsequent content in the films was 500 ppm (0.05 %) and 1,000 ppm (0.1 %) respectively.

Next, a twin-screw extruder ZSK 25 (manufacturer: Coperion GmbH in Stuttgart, Germany) was used at 265 °C to produce flat films with a thickness of about 150 µm. Samples measuring 85 mm x 85 mm were cut out of the film and stretched biaxially (simultaneous lengthwise and crosswise stretching in the film's direction of travel). With an oven temperature of 90°C, and a 50-second pre-heating time, the films were stretched at a speed of 100 %/s up to a ratio of 3,5 x 3,5. No subsequent thermosetting was carried out. The resulting final thickness of the film was about 15 µm.

The Result

Evaluation of slip-stick behavior was done by determining the coefficient of friction (COF). The lower the COF value, the better will the film slide over the selected substrate. The COF of film/metal is an indication of how the film will behave while being processed in fast-running packaging machines.

In the case of Silfit Z 91 and the two precipitated silicas, the addition of mineral additive results in a significant dosage-dependent reduction of the film/metal friction coefficient. Hereby, Silfit Z 91 achieves similar slip-stick properties as the precipitated silicas. In contrast, pyrogenic silica is clearly far less effective. Also the higher dosage of 1,000 ppm only had a limited influence on the film/metal COF. This result could also be observed for the dynamic film/metal COF (**Fig. 2**).

Based on the film/film COF it is possible to assess the film's behavior while processing film rolls. The lower the COF, the lower is the tendency for individual film layers to adhere to each other on the roll. Measurement of the film/ »

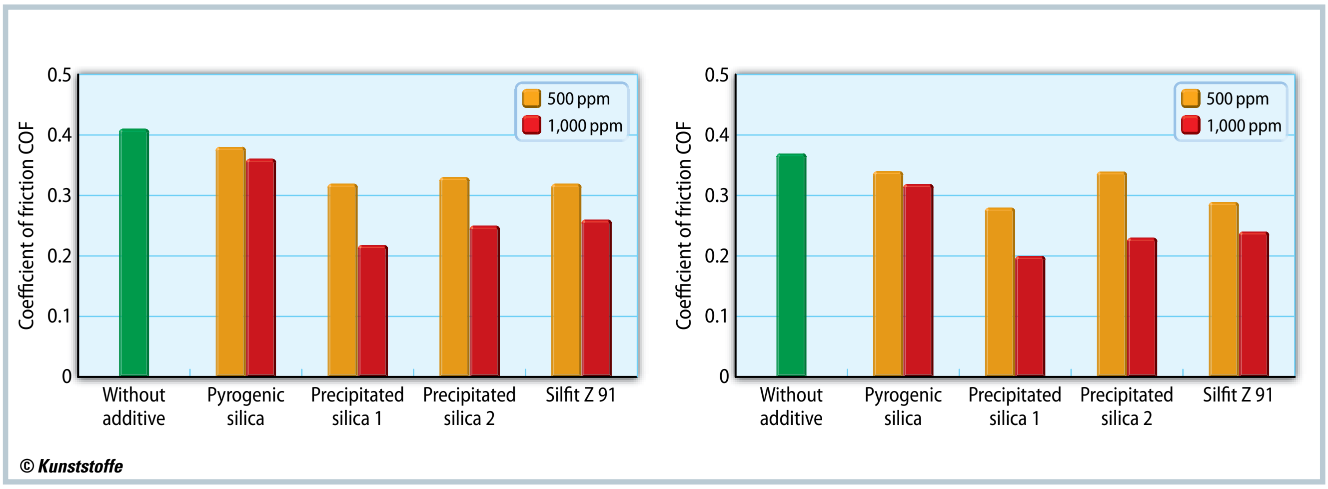

Fig. 2. Comparison of friction coefficients of mineral additives: Film/metal, static (left) and film/metal, dynamic (right)

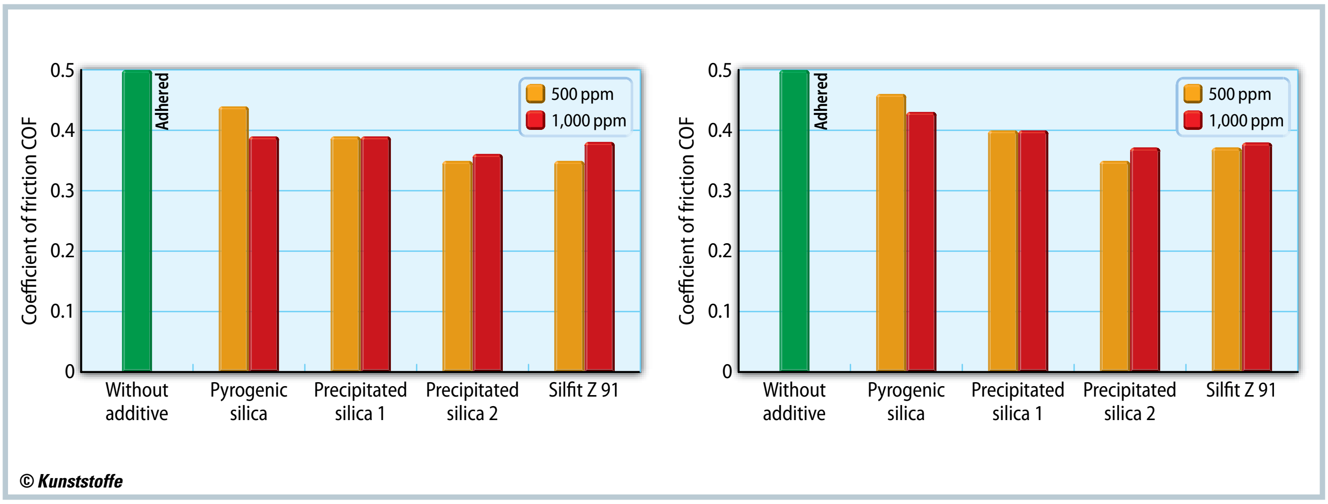

Fig. 3. Comparison of the COF for mineral additives: Film/film, static (left) and film/film, dynamic (right)

film COF was practically impossible on the samples without mineral additive, because the film sections adhered too strongly (blocking). With the addition of mineral additive, the film/film COF drops significantly, but a dependence on the dosage could not be determined. Silfit Z 91 lies at least at the same level as the considerably more expensive silicas (**Fig. 3**).

When the films are used in the packaging industry, good optical properties such as high gloss and transparency as well as the lowest possible haze values are usually expected. Film gloss was measured with an incidence angle of 45°. Through the use of mineral additives, and depending on the dosage, film gloss is slightly reduced. Hereby, practically no differences were observed between the used additives (**Fig. 4**).

Transmittance defines the ratio between transmitted light and incident light. Its value can be reduced by absorption and reflection. Films with about 90 % transmission are considered to be crystal clear. No significant de-

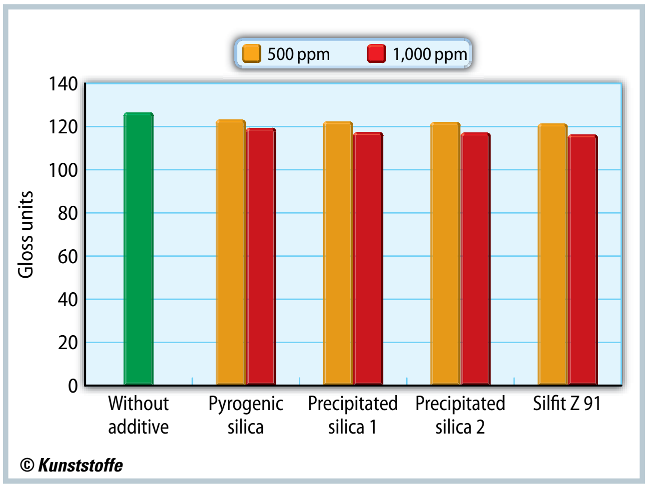

Fig. 4. Comparison of gloss 45° values of the mineral additive

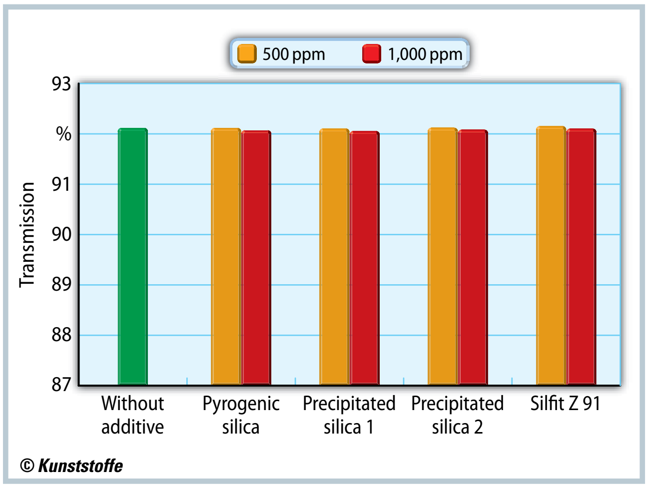

Fig. 5. Comparison of transmittance values of the mineral additive

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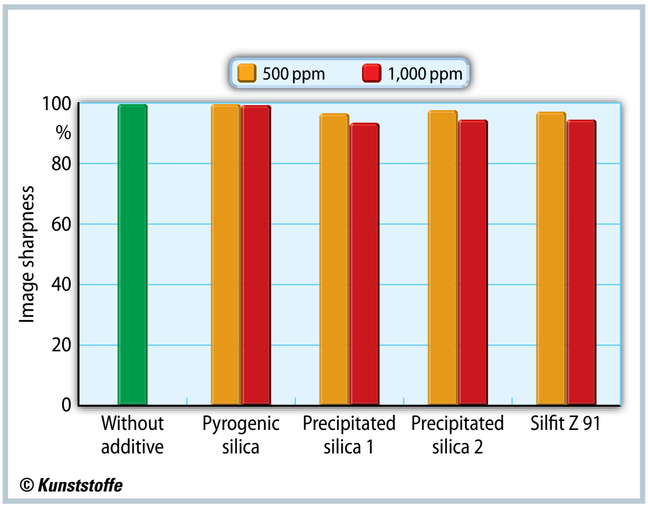

Fig. 6. Comparison of values for image sharpness (clarity) of the mineral additive

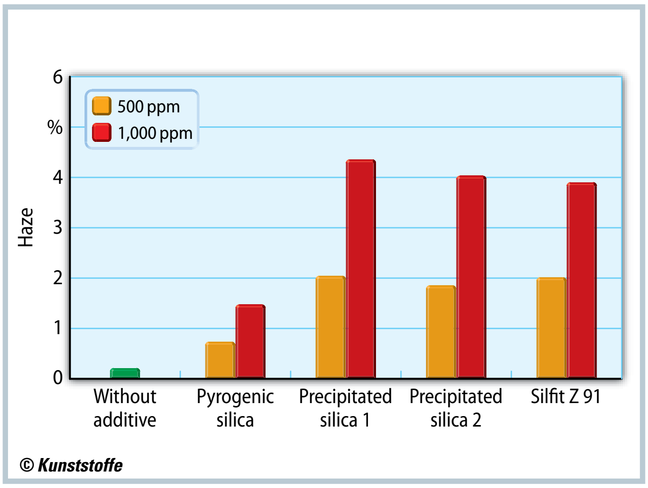

Fig. 7. Comparison of haze values of the mineral additive

pendency was observed here, neither from the additive used nor from the dosage. The values obtained are at the same level as for films without mineral additive (**Fig. 5**).

The amount of transmitted light is divided into directed and diffuse portions. In order to evaluate image sharpness (clarity), the diffuse proportion (scattered light) is observed in an angular range of <2,5° (small-angle scattering). Due to this scattered light, contours become distorted, and appear less sharp. The higher the value, the sharper is the image when viewed through the film. As opposed to pyrogenic silica, a low reduction of image sharpness is exhibited by the two precipitated silicas and Silfit Z 91 (**Fig. 6**).

Film haze can be assessed by means of the amount of scattered light in an angular range of >2.5° (wide-angle scattering). The higher the haze value, the milkier is the film's appearance, and the lower are contrast, transparency, and gloss. Similar to gloss, film haze is also influenced noticeably by mineral additives, and a dependency on dosage is also observed. The best value achieved by the pyrogenic silica used here was only 0.7 or 1.5 %. However, this is put into perspective by the comparatively weak results for the coefficient of friction. Precipitated silicas have haze values that are approximately twice as high, but exhibit better friction coefficients than the pyrogenic version. Hereby, Silfit Z 91 is fully comparable with precipitated silicas (**Fig. 7**).

In a price comparison of the purely mineral additive, Silfit Z 91 only costs about 1/10 of the silicas normally used.

Summary

Compared with antiblock additives based on synthetic silicas, the properties profile of Silfit Z 91 is similar to that of precipitated silicas. In addition to a low coefficient of friction, good optical properties are obtained.

Thanks to the high bulk density, dust generation is minimal. As a mineral additive, Silfit Z 91 is easily dispersible. Because of the low BET surface area, interactions with other additives can be practically excluded.

Compared with synthetic silicas, which are often used for optically demanding films, there is a significant cost advantage. On the whole, and even with low dosage, a good antiblocking effect is achieved, combined with only minimum impairment of the optical properties. Therefore, Silfit Z 91 is very well suited as a cost-effective antiblock additive for PET films. ■