

# Martinal LEO: New aluminium hydroxides with improved performance

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*Aluminium hydroxide (ATH) is well known as an environmentally friendly mineral flame retardant for plastics and rubber. Large amounts of finely precipitated ATH grades are used in the wire and cable industry. Some traditional products, however, show some drawbacks both during processing and in certain usage properties of the final flame retarded product. This paper describes in detail the benefits of newly developed fine ATH grades, which not only allow for a more efficient handling of the powder itself but also a higher throughput during compounding and subsequent processing steps. Additional advantages like better thermal stability, a lower viscosity of the filled compound and better electrical properties for cable applications will be illustrated.*

## 1. Introduction

Aluminium hydroxide (ATH) is used in large quantities in plastics, rubber, latices and in paper applications as environmentally friendly, non toxic flame retardant. The flame retardant effect is mainly due to the endothermic decomposition of the hydroxide  $\text{Al}(\text{OH})_3$  into the thermally stable oxide  $\text{Al}_2\text{O}_3$  at a temperature of about 200–230 °C. The released water vapor dilutes the burnable gases and oxygen, and an aluminium oxide layer is formed which protects the undamaged plastic. Loadings of about 50 to 65 wt.-% are necessary to pass relevant fire standards. Such high amounts of filler dilute the compound, which leads to less fuel available that can feed the flame. In plastics that generate a lot of smoke during burning, like PVC-P, ATH acts not only as a flame retardant, but also as a smoke suppressant and reduces the smoke density significantly.

One basically distinguishes between coarser ATH grades and finely precipitated products. The coarse ATH types with a  $d_{50}$  value

between about 60 and 6  $\mu\text{m}$  are mainly used in thermosets and latices, sometimes in combination with finely precipitated grades. The  $d_{50}$  of the latter lay typically between about 0.7 and 2.5  $\mu\text{m}$ . A major field of application for the finely precipitated ATH is the wire and cable industry. These products are used in insulation as well as in sheathing compounds, while the coarser grades are more common in bedding compounds.

Handling and incorporation in plastics of solid particles being about 100 times smaller than a human hair can be somewhat delicate due to the high loadings, especially at highest throughputs during compounding. For an economic production of the intermediate and final products, the processability of the compound, for example during extrusion, is an important property. A key factor besides the quality of the surface of the extrudate is the viscosity of the compound: higher viscosities inevitably reduce the throughput, because the higher friction leads to higher temperatures which in turn cause an early decomposition of the ATH into the oxide phase. A new aluminium hydroxide grade with a higher thermal stability that would additionally result in lower compound viscosities is a desirable product enhancement.

For cable insulation compounds, a high electrical resistivity is important, especially after ageing processes in water. So far, in particular the finer ATH grades with a higher

specific BET surface, had problems to fulfill this requirement: the electrical resistivity of the resulting insulation compounds was inadequate to pass relevant standards.

The following paper explains the advantages of newly developed, finely precipitated ATH grades that largely overcome the above mentioned drawbacks.

## 2. Common finely precipitated ATH grades

The market offers finely precipitated ATH fillers with different specific BET surfaces. One reason is that the mechanical properties can be optimised as a function of the BET surface: the higher the specific surface, the higher the tensile strength of the plastic compound. This relationship is also well known from other fillers like carbon black and silica [1]. **Figure 1a** shows this effect exemplarily for a plastic compound, consisting of 40 wt.-% EVA (Scorene Ultra UL00119), 60 wt.-% ATH with different BET surfaces, 0.8 wt.-% (relative to the filler) of the amino silane Dynasylan AMEO and 0.75 wt.-% of the antioxidant Ethanox 310. The fillers Martinal OL-104 LE, Martinal OL-107 LE and Martinal OL-111 LE have a specific BET surface of 4, 7 and 11  $\text{m}^2/\text{g}$  respectively. The indicated BET values are the mean values of the specified range and the abbreviation "LE" means "Low Electrolyte". These ATH grades thus contain low amounts of electrolytes.

Products with a higher specific surface also lead to a higher oxygen index (LOI). Quite often, the oxygen index is used to assess flame retardancy: the higher the LOI value, the better the compound is flame retarded. This interrelationship is shown in **figure 1b** for the plastic compound with the composition given above.

Due to the higher specific surface, these fillers are generally finer, or more precisely, they have a finer particle size distribution. A characteristic quantity of the particle size distribution is the median value  $d_{50}$ . The median value expresses that 50 % of the particles are smaller and bigger respectively. Laser scattering is a common method used to measure particle size distributions.

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### 3. Different powder properties of the new LEO products

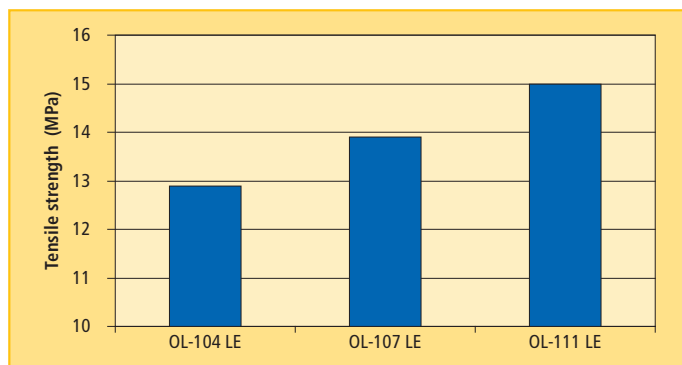
**Table 1** contains the most important powder properties of the traditional "LE" and the new "LEO" products. The "O" in "LEO" means "optimised".

The high aluminium content of 99.4 % and the typical value for the glow residue of 34.5 % suggest that the listed products

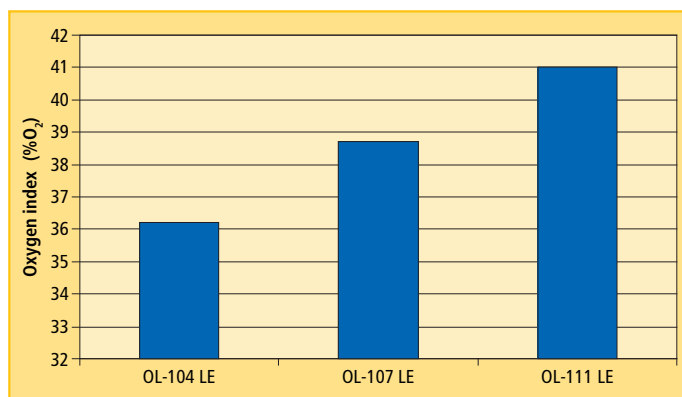
are not coated and that the crystal structure has not been modified. The surface moisture, measured by the difference in weight before and after 4 h drying in an oven, is also unchanged between the LE and LEO products having the same BET. There is also no difference between the range of the BET surface, the refractive index, the whiteness and the density. It is remarkable, however, that the range for the  $d_{50}$  value has become much smaller for both the Martinal OL-104 LEO and the Martinal OL-107 LEO. While for the

traditional products, the range was 1  $\mu\text{m}$  and 0.6  $\mu\text{m}$  respectively, for the LEO grades it could be reduced to 0.4  $\mu\text{m}$  and 0.3  $\mu\text{m}$ . For comparison: such small tolerances for the  $d_{50}$  value lay in the order of magnitude of the diameter of bacteria! For the end user, this implies a much more constant product quality, resulting in more constant production processes and final product properties.

For Martinal OL-107 LEO, it is particularly noticeable that beside a reduction of the  $d_{50}$  range by a factor of 2, the level of the  $d_{50}$  is much higher as for the corresponding traditional LE product and it overlaps with the  $d_{50}$  range of the Martinal OL-104 LEO. This means that it was possible to develop a new product with a higher specific BET surface that offers advantages regarding tensile strength and oxygen index (see **fig. 1a** and **b**) while keeping the particle size on the level of a typical BET= 4  $\text{m}^2/\text{g}$  product. This new product property has far-reaching consequences for the bulk density, the powder flowability and the rheological properties of the plastic compound, as will be shown in the following sections.



**Fig. 1a:** Influence of the BET-surface on the tensile strength of a plastic compound



**Fig. 1b:** Influence of the BET-surface on the oxygen index of a plastic compound

**Tab. 1:** Powder properties of the traditional „LE“ and the new „LEO“ products

		Martinal OL-104 LE	Martinal OL-104 LEO	Martinal OL-107 LE	Martinal OL-107 LEO
Al(OH) <sub>3</sub> -content	(%)	99.4	99.4	99.4	99.4
Glow residue at 1200 °C	(%)	≈ 34.5	≈ 34.5	≈ 34.5	≈ 34.5
Moisture after 4 h oven drying at 105 °C	(%)	≤ 0.35	≤ 0.35	≤ 0.4	≤ 0.4
Specific surface (BET)	( $\text{m}^2/\text{g}$ )	3 – 5	3 – 5	6 – 8	6 – 8
Refractive index		1.58	1.58	1.58	1.58
Whiteness (Elrepho)	(%)	≥ 94.0	≥ 94.0	≥ 94.0	≥ 94.0
Density	( $\text{g}/\text{cm}^3$ )	2.4	2.4	2.4	2.4
Median particle size $d_{50}$ (Cilas laser scattering)	( $\mu\text{m}$ )	1.3 – 2.3	1.7 – 2.1	1.1 – 1.7	1.6 – 1.9
Thermal stability (TGA at 2 % weight loss, 1 K/min)	(°C)	≈ 220	≈ 225	≈ 207	≈ 220
Electrical conductivity (10 % in H <sub>2</sub> O)	( $\mu\text{S}/\text{cm}$ )	≤ 60	≤ 60	≤ 150	≤ 70
Oil absorption	( $\text{ml}/100 \text{ g}$ )	28 – 35	27 – 32	29 – 37	28 – 33
Na <sub>2</sub> O soluble	(%)	≤ 0.08	≤ 0.08	≤ 0.1	≤ 0.08

Both LEO products significantly improved their thermal stability according to **table 1** by 5 °C for the Martinal OL-104 LEO and by about 13 °C for the Martinal OL-107 LEO (measured at a TGA weight loss of 2 % and using a temperature gradient of 1 K/min). Also remarkable is the electrical conductivity of the Martinal OL-107 LEO being about 50 % lower in a 10 % aqueous suspension than for the traditional LE grade. The advantages of these additional product enhancements will also be explained in the following sections.

### 4. Improved bulk density and flowability of the new LEO grades

The bulk density and the flowability of bulk material, especially of fine powders, depends on the history of the material. It is obvious that mechanically compacted fillers show a better flowability due to the higher density of the compressed powder. Mechanical compacting is thus an option to improve both bulk density and flowability.

Very often, however, after having been compacted, fillers have to be conveyed again, for instance into a small silo or from a big bag to the compounding unit. Thereby, the artificially improved bulk density gets reduced or even completely destroyed. For bulk deliveries in silo trucks, conveying is necessary before and after truck loading, which makes mechanical compaction completely useless. A filler showing an intrinsically improved bulk density and flowability behavior after conveying processes would thus be preferable and would have considerable advantages versus traditional products. **Table 2** shows that this is indeed the case for the new LEO types. First, the products were taken from the packaging without any further mechanical treatment. The bulk density was measured by weighing the powder in a 50 cm<sup>3</sup> metal cylinder. The time necessary for 100 g of the powder to flow through a vibrating funnel (amplitude: 0.5 mm at constant frequency, testing apparatus from the Retsch company) was used to assess powder flowability. The faster the sample flows through the funnel, the better the flowability of the powder.

The data of the untreated samples in **table 2** show that the bulk density and the flowability of the new LEO grades are higher compared to the traditional LE types. In order to simulate a pneumatic conveying process, 1 kg of ATH was fluidized during 10 min at 1000 rpm in a high shear Henschel mixer (type FM 10 C). Immediately after the mechanical treatment, the bulk density and the flowability were measured as described above. The bulk density and flowability were significantly reduced, as expected. For the new LEO products, however, the obtained bulk density is not only much higher than for the corresponding LE grades with the same BET surface, but almost on the same level. This results in additional advantages for the packaging units: the Martinal OL-107 LEO can be packed in 25 kg bags and in 1000 kg big bags as the Martinal OL-104 LEO, which was impossible with the Martinal OL-107 LE due to its lower bulk density. The loading of silo trucks can be increased accordingly. The benefits for the customer are obvious: less space is required in the warehouse and more ATH can be filled into a silo. The higher bulk density after conveying processes also offers

advantages for compounding with internal mixers: due to the smaller ATH volume for a given mass, the free volume of the mixing chamber is higher and offers the possibility to increase the batch size and thus the throughput [2].

The higher bulk density and the better flowability after conveying also result in a better compounding performance in continuous mixers like in Buss Ko-kneaders, as will be shown in section 7.

### 5. Better thermal stability

The major flame retardant effect of ATH is based on the endothermic decomposition of the hydroxide into the oxide phase. The measured value for the start of the decomposition depends on the crystallite size and also on the test parameters. Coarser ATH types can have a higher thermal stability by approx. 10 °C than fine grades. Higher temperature gradients during TGA testing also resulted in apparently higher values for the thermal stability because the sample, due to its low thermal conductivity (especially at higher net weight), has not yet adapt-

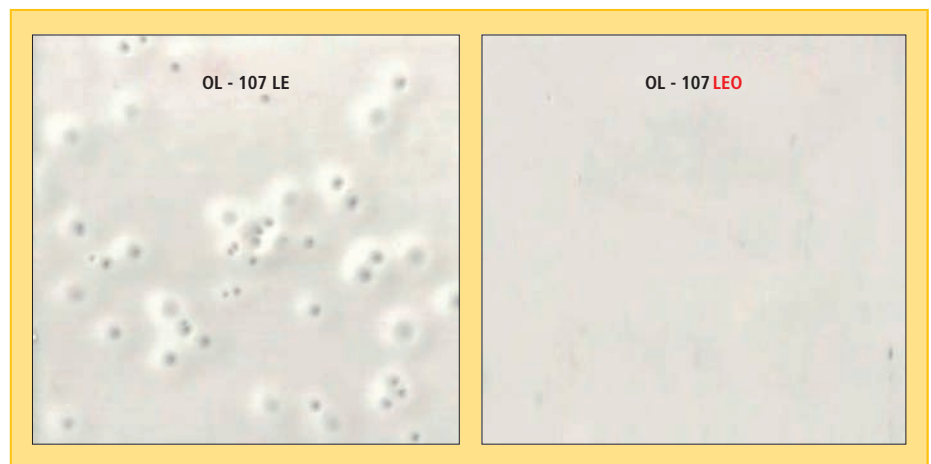
ed the displayed temperature. This explains some considerable differences between figures found in literature. The data listed in the following table have been measured using a temperature gradient of 1° K/min. Due to the normal surface moisture of the sample as indicated in **table 1**, the error of the temperature for the 1 % weight loss value is somewhat higher. Therefore, the 2 % weight loss data are also listed in **table 3**.

A significant improvement of the thermal stability by about 7 °C was obtained for the new Martinal OL-104 LEO (at 2 % weight loss) and an even more pronounced increase for the Martinal OL-107 LEO by about 13 °C.

In order to depict the improved thermal stability in a plastic compound, 60 wt.-% of Martinal OL-107 LE and Martinal OL-107 LEO were mixed into 40 wt.-% of EVA (Escorene Ultra UL00119) and a 1 mm thick plate was made by compression moulding while keeping a temperature of 195 °C during 2 min. **Figure 2** shows the result.

In contrary to the plate filled with Martinal OL-107 LEO where only a few blowholes

**Fig. 2:** Effect of the improved thermal stability of the LEO products



**Tab. 2:** Bulk density and flowability of the traditional „LE“- and the new „LEO“ products

		Martinal OL-104 LE	Martinal OL-104 LEO	Martinal OL-107 LE	Martinal OL-107 LEO
Bulk density	(g/cm <sup>3</sup> )	378	498	267	367
Flowability (explanation see text)	(s)	19	12	18	14
Bulk density after 10 min at 1000 rpm in a Henschel mixer	(g/cm <sup>3</sup> )	272	318	180	314
Flowability after 10 min at 1000 rpm in a Henschel mixer	(s)	33	21	35	26

appear, intensive blistering in the plate filled with Martinal OL-107 LE becomes visible due to the early decomposition of the traditional Martinal OL-107 LE product. An improved thermal stability allows for higher temperatures during compounding and extrusion, which in turn positively affects throughput and operating efficiency.

## 6. Better electrical properties

The use of ATH as a flame retardant in cable insulation compounds is a demanding application due to the required high electrical resistivity of the filled compound. Relevant cable standards require for example a minimum value after ageing in liquid media for a certain time at a certain temperature. **Table 4** shows exemplarily the electrical resistivity and the water absorption in wt.-%

of a 2 mm thick plate for the LE and the LEO products after 7 days ageing in deionized water at 70 °C. The formulation used is also given in **table 4**.

One can see that the electrical performance and the water absorption were significantly improved, both for Martinal OL-104 LEO and Martinal OL-107 LEO.

## 7. Improved compounding performance

Depending on the fire standard the finished product needs to pass and the plastic or rubber compound involved, typical ATH loadings between 50 and 65 wt.-% are to be incorporated. Internal mixers are used quite often to accomplish this, and the advantages of the new ATH products in these type of

mixing machines due to the higher bulk density and flowability have already been discussed in sections 4 and 5. A considerable improvement is also obtained for continuous mixers, as will be shown exemplarily for a Buss Ko-kneader.

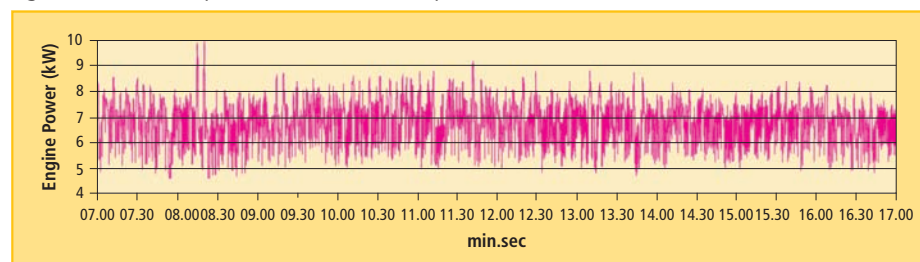
The characteristic feature of Buss Ko-kneaders is their principle of mixing [3], allowing for highly filled compounds to be produced with a very good filler dispersion. For reasons of operating efficiency, it is common to drive the throughput to the limit of the temperature stability of the compound, which, in most cases, is predetermined by the thermal stability of the ATH. Also here, the new LEO products offer advantages due to their higher thermal stability. At very high throughputs, however, another phenomenon appears for the traditional products which leads to process variations and thus has a negative impact on the throughput: high variations of the engine output of the mixing screw motor and/or the discharge extruder. **Figure 3a** and **b** show the power consumption of the motor of a 46 mm Buss Ko-kneader as a function of the mixing time for an EVA/PE compound filled with 65 wt.-% of Martinal OL-104 LE and Martinal OL-104 LEO respectively. A maleic anhydride grafted polymer was used as a polymer-filler coupling system and the throughput was set to 25 kg/h.

One can clearly see the smaller variations for the compound filled with Martinal OL-104 LEO. In order to compare the results quantitatively, the standard deviation was calculated from the recorded power data. For the curves in **figure 3a** and **3b**, a value of 0.87 kW and 0.54 kW was found respectively, the latter being significantly lower.

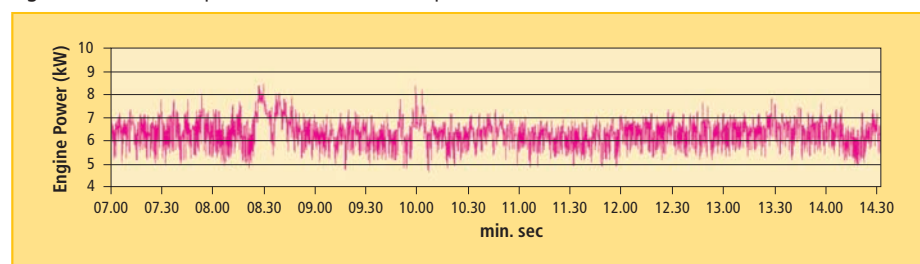
The trial was repeated with Martinal OL-107 LE and Martinal OL-107 LEO, now using an EVA/PE compound at 60 wt.-% filler loading and an amino silane as a coupling agent. The results are shown in **figure 4a** and **b**.

In this formulation, the Martinal OL-107 LEO results in smaller variations of the power consumption. The standard deviation was 0.41 kW for the Martinal OL-107 LE and 0.33 kW for the Martinal OL-107 LEO. It is remarkable to note that the level of the power

**Fig. 3a:** Power consumption of the motor for a compound filled with Martinal OL-104 LE



**Fig. 3b:** Power consumption of the motor for a compound filled with Martinal OL-104 LEO



**Tab. 3:** Thermal stability of the traditional „LE“- and the new „LEO“-products

		Martinal OL-104 LE	Martinal OL-104 LEO	Martinal OL-107 LE	Martinal OL-107 LEO
Temperature at 1 % weight loss	(°C)	205	209	195	206
Temperature at 2 % weight loss	(°C)	219	226	207	220

**Tab. 4:** Electrical resistivity after water ageing of the „LE“- and the new „LEO“-products

		Martinal OL-104 LE	Martinal OL-104 LEO	Martinal OL-107 LE	Martinal OL-107 LEO
Electrical resistivity after 7 days in 70 °C hot water	(Ω cm)	$2 \cdot 10^8$	$4 \cdot 10^{10}$	$3 \cdot 10^7$	$2 \cdot 10^{10}$
Water absorption	(%)	4.8	2.9	10.7	3.3
Formulation: EVA (28 % VA): 80 phr · LLDPE: 20 phr · ATH: 150 phr · Antioxidant: 0.75 phr					

curve is lower. While the mean value for all the data shown in **figure 4a** was 6.5 kW, it was only 5.9 kW for the data in **figure 4b**. The reason is the reduced compound viscosity for the LEO products. This particular property will be discussed in more detail in the next section.

### 8. Lower compound viscosity during processing and typical property profile of two cable compounds

Thermoplastic cable compounds flame retarded with ATH generally consist of an EVA/PE blend, ATH, a coupling system, antioxidants and eventually processing aids. The coupling system provides a link between the filler and the polymer and also reduces the filler-filler interaction. At these high filler loadings, coupling systems are necessary to obtain good mechanical, rheological and (in case of insulation compounds) electrical properties. **Table 5** shows 2 simple cable formulations. In formulation 1, a vinyl silane/peroxide coupling system was chosen, and in formulation 2 a polymeric system with maleic anhydride.

The compounds were mixed on a two-roll mill and the data in **table 6** were measured according to the corresponding ISO standards.

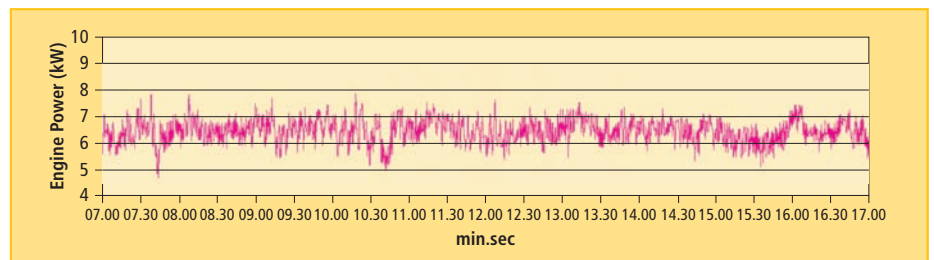
While there is no significant difference in these 2 formulations between the mechanical properties and the oxygen index, the melt flow index is significantly higher for the Martinal OL-104 LEO and the Martinal OL-107 LEO grades compared to the traditional ATH products, especially for the Martinal OL-107 LEO: despite the higher specific BET surface, the obtained viscosities are in the typical range for a 4 m<sup>2</sup>/g product. This offers new possibilities for the end user to optimize his product performance: the higher specific surface allows for higher tensile strength and LOI values while maintaining good rheological properties. As shown in the sections above, there are no more drawbacks regarding bulk density, flowability, electrical resistivity and compounding performance, as it was the case for the Martinal OL-107 LE.

Because the melt index is measured at one shear rate only, additional trials have been made with formulation 1 on a capillary rheometer at 190 °C. **Figure 5** shows the results. The improved rheological behaviour of the new LEO products was again confirmed for a broad range of shear rates.

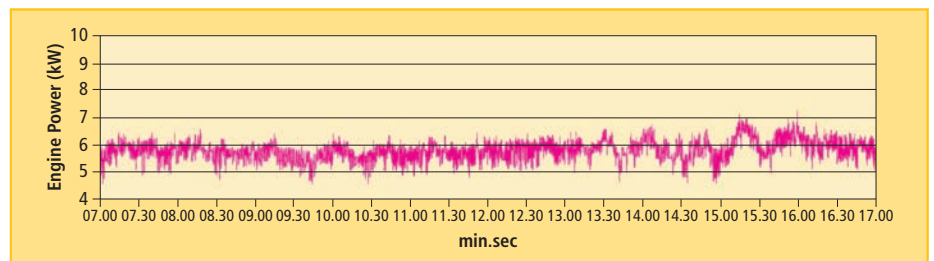
### 9. Flame retardant properties: Cone data and cable bundle flame propagation test

For the investigated formulations, according to **table 6**, no significant differences were found for the oxygen index and thus

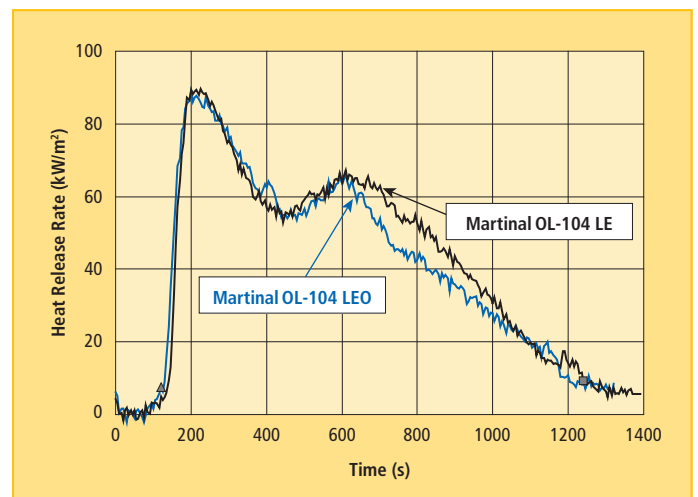
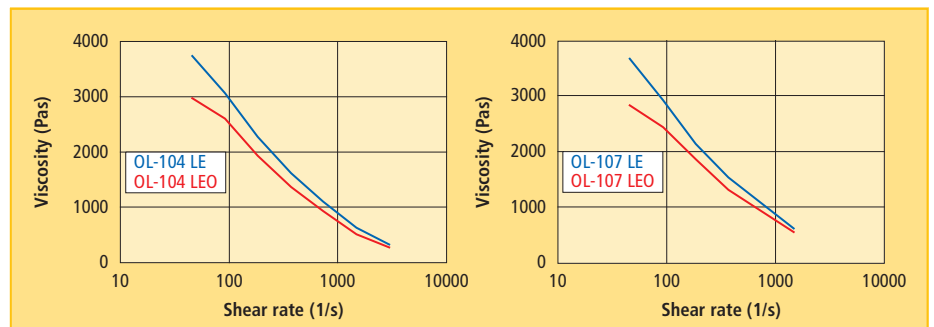
**Fig. 4a:** Power consumption of the motor for a compound filled with Martinal OL-107 LE



**Fig. 4b:** Power consumption of the motor for a compound filled with Martinal OL-107 LEO



**Fig. 5:** Viscosity function for the traditional and new ATH grades in formulation 1



**Fig. 6:** Cone calorimeter results for Martinal OL-104 LE and Martinal OL-104 LEO using formulation 1

for the fire performance between the traditional LE and the new LEO products with the same specific BET surface. As is generally known, an assessment of the fire performance based on the oxygen index alone is rather limited. A laboratory technique with a better informational value is the measurement of the heat release rate (HRR) in a cone calorimeter. In this section, the flame retardant efficiency of Martinal OL-104 LE and Martinal OL-104 LEO will be compared using formulation 1 (see **tab. 5**). In addition, the result of a flame propagation test according to IEC 60332-3 cat. C on a NHXMH cable bundle will be presented.

Cone calorimeter testing was performed according to ISO 5660-1 on 3 mm thick compression moulded plates and a heat radiation of 35 kW/m<sup>2</sup>. As shown in **figure 6**, the two heat release rate curves do not show a significant difference for either the time to ignition (TTI) value or for the peak heat release rate (PHRR).

In order to investigate the fire performance on a final good, a NHXMH installation cable as shown in **figure 7** was produced and tested according to IEC 60332-3 cat. C.

These cables are made of a halogen free flame retarded, thermoplastic sheathing compound and the copper wires are insulated with cross-linked polyethylene. A three-conductor cable was chosen with a wire cross section of 1.5 mm<sup>2</sup>. The wires are embedded with a bedding compound. By default, the sheathing compound is flame retarded with Martinal OL-104 LE and this cable is known to pass the flame propagation test according to IEC 60332-3 cat. C. In order to investigate the flame retardant properties of Martinal OL-104 LEO, the LE product was simply replaced by the LEO grade. **Figure 8** shows the cable tray in the furnace chamber. The cables are mounted on a testing frame, which is fixed vertically on the rear panel of the furnace chamber. For a simulation of a realistic fire scenario, an air stream of 5 m<sup>3</sup>/min enters at the bottom and flows through the chamber. A propane gas burner ignites the cable bundle during 20 min at a distance of 75 mm. The test is to be classified as passed if the flames self-extinguish and if no fire damage at the front or rear side of the cable bundle can be found 250 cm above the lower edge of the burner. In the present case, the burned length was found to be 195 cm and the test was thus passed without

any problems. Although these results suggest that in most cases the LEO grades can be used as a drop in solution for other ATH grades currently being used, this has to be verified case by case.

## 10. Summary

The new developed aluminium hydroxide flame retardants Martinal OL-104 LEO and Martinal OL-107 LEO offer many advantages to the end user due to their improved product properties. The bulk density and the flowability of the powder, especially after conveying processes, are significantly higher. The better thermal stability of the new aluminium hydroxides allows for higher processing temperatures both during compounding and extrusion, which in turn allows for higher throughput. In continuous mixers like for example Buss Ko-kneaders, the faster wettability of the filler with the polymer results in less variations of the engine power consumption and also improves the constancy of the compound at even higher throughputs. Another benefit, especially for cable insulation compounds, is the improved electrical performance, mainly for

	Formulation 1	Formulation 2
	(phr)	(phr)
EVA, 28 % VA	80	67
mLLDPE	20	–
LLDPE	–	17
ATH	170	160
Vinyl silane	1	–
Peroxide	0.07	–
Maleic anhydride coupling system	–	16
Primary antioxidant	0.75	0.75

Tab. 6: Compound data of the formulations in Table 5

		Martinal OL-104 LE	Martinal OL-104 LEO	Martinal OL-107 LE	Martinal OL-107 LEO
<b>Formulation 1</b>					
Tensile strength	(MPa)	11.9 ± 0.2	11.8 ± 0.2	14.3 ± 0.2	14.1 ± 0.2
Elongation at break	(%)	185 ± 5	183 ± 5	157 ± 5	163 ± 5
Melt flow index, MFI 150 °C	(g/10 min)	0.3 ± 0.1	0.6 ± 0.1	0.1 ± 0.05	0.4 ± 0.1
Oxygen index LOI	(% O <sub>2</sub> )	33.8 ± 0.5	33.7 ± 0.5	34.3 ± 0.5	34.3 ± 0.5
<b>Formulation 2</b>					
Tensile strength	(MPa)	11.0 ± 0.2	11.1 ± 0.2	13.1 ± 0.2	13.0 ± 0.2
Elongation at break	(%)	208 ± 5	205 ± 5	202 ± 5	217 ± 5
Melt flow index, MFI 150 °C	(g/10 min)	4.4 ± 0.2	4.8 ± 0.2	2.8 ± 0.2	4.6 ± 0.2
Oxygen index LOI	(% O <sub>2</sub> )	35.2 ± 0.5	35.1 ± 0.5	37.1 ± 0.5	37.3 ± 0.5

Tab. 5: Typical cable formulations, flame retarded with ATH

Fig. 7: NHXMH-cable for the flame propagation test

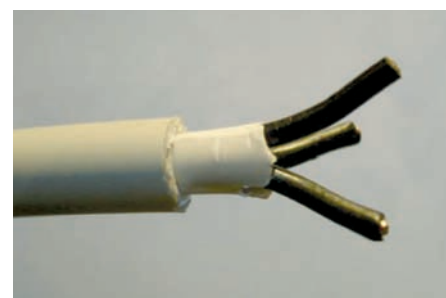


Fig. 8: Cable flame propagation test according to IEC 60332-3 cat. C



the Martinal OL-107 LEO compared to the former Martinal OL-107 LE product. In addition, it was possible to further optimize the ATH fillers so that the viscosity of the filled compound gets reduced, an advantage that takes effect e.g. during extrusion processes. Remarkably is also the fact that the Martinal OL-107 LEO, despite of its higher specific BET surface, has a particle size distribution comparable to the coarser Martinal OL-104

LEO. In combination with the other improved properties mentioned above, the end user for the first time has the possibility to take advantage of the higher specific surface for compound optimisation without compromising in powder bulk density and flowability, electrical properties, thermal stability, rheological properties and wettability during mixing as it was the case for other traditional products like Martinal OL-107 LE.

## 11. Literature

- [1] W. Kleemann, „Mischungen für die Elastverarbeitung“, VEB Deutscher Verlag für Grundstoffindustrie, Leipzig, 1982, p. 132 ff.
- [2] R. Herbiet, GAK 53 (March 2000), 194
- [3] R. H. Wildi, Ch. Maier, “Understanding Compounding”, Hanser Publishers, Munich, 1998, p. 54 ff.

## Choosing the right extensometer

The requirements for an extensometer are determined primarily by the characteristics of the material to be tested. This includes its shape and dimensions, test requirements, and the formal standards which must be met. These define the gauge length, accuracy, test sequence, and environmental conditions. The right choice of extensometer cannot be limited to the basic material characteristics such as specimen dimensions, stiffness, strength and plasticity alone. It is also necessary to decide whether an extensometer can be connected directly to the specimen without influencing the load measurement or mechanically damaging the specimen itself. Very thin specimens such as foils can be sensitive to clamping forces, whilst very small wire specimens do not provide enough visible area for reliable non-contact measurements. A high stiffness in the initial extension range, followed by high plasticity traditionally requires more than one extensometer. The first meas-

ures small strains (typically up to 5 mm) very accurately in the elastic range, and the second measures very high extensions (typically  $\geq 500$  mm). Specimen with very smooth surfaces, or made of transparent materials are not suitable for non-contact measurements without first fixing measuring marks onto the surface of the specimen (**fig. 1**).

One very important consideration is the behaviour when the specimen fails. Metals and hard plastics will slip through the knife edges of a contact extensometer without damaging them, and rotatable knife edges should be used to further reduce the risk of damage even if the surface of the specimen is particularly rough. High extension or flexible specimens can damage or destroy the knife edges and even the extensometer itself due to whiplash, splintering, or de-lamination of specimens (for example steel rope). For these applications non-contact measurement is a must.

With contact type measuring extensometers, the measurement travel is normally an engineered and fixed value which is dependent upon the range of the measurement transducer and, with fulcrum hinged sensor arms, the leverage ratio. The initial gauge length is set manually with fixed steps or automatically over a defined range. Non-contact extensometers that use a video camera must have the field of view larger than the required range plus the initial gauge length. Since the specimen portions which are outside the gauge length, and the machine components themselves deform in the direction of loading, the position of the measuring marks on the specimen changes during the test. For extension and/or gauge lengths which are expected to be outside of the field of view then the objective lens must be changed or the distance between the specimen and the video camera must be increased. All these actions decrease the measuring accuracy, and in addition, every changed measurement configuration must be adjusted and calibrated.

Devices which are easy to set-up and sequences which can be automated reduce

Fig. 1: Schematic diagram showing the operating principle of various extensometers

