ACTILOX® PA-B2

Processing Aid and Flame Retardancy Booster

A novel mineral based flame retardant synergist for Wire & Cable

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ACTILOX® PA-B2

Metal hydrates, simply called mineral flame retardants, are the most important group of flame retardants used in wire & cable applications. Polyolefin compounds highly filled with aluminium hydroxide (ATH) or magnesium hydroxide (MDH) are known as Halogen-Free-Flame-Retardant (HFFR) compounds and are widely used for sheathings of electrical and communication cables. However, for Low Fire Hazard and Fire Resistant cables most stringent requirements are imposed. Those standards are difficult to meet and frequently render the additional incorporation of flame retardant synergists necessary.

Known synergistic flame retardants like organically modified nanoclays very often impact compound processability and aging performance negatively. This paper will demonstrate how these issues can be overcome by using **ACTILOX® PA-B2**, focussing on processing, fire retardancy, mechanical and aging performance.

- **Product properties**
  - High temperature stability
  - Excellent polymer compatibility
  - Easy blendable with mineral fillers
- **Compound properties**
  - Synergistic effects with ATH
  - Reduced pressure build-up
  - Improved processability compared to other synergists
  - Improved hot air aging performance
  - No negative impact on mechanical properties
  - No need for expensive stabilizer/antioxidant packages

1. Introduction

Fire safety is a big challenge which is getting more and more important due to the continuously increasing amount of plastic products in our daily lifes. In the past, major fires have caused heavy loss of life which shows that fire standards need to be constantly optimized and adapted to current circumstances. One example of fire standards getting more consistent and stringent is the Construction Product Regulation (CPR, EU/305/2011) amendment from 2017. This regulation determines that all installation cables have to be treated as construction products and thus comply with the requirements given in the new harmonized standard EN 50575. This standard specifies the requirements for the fire performance of cables which are used as permanent systems in buildings or tunnels. This includes power cables, control and communication cables as well as fiber optic cables.

According to CPR and the standard EN 50575 seven Euroclasses exist classifying the reaction of cables to fire (Aca, B1ca, B2ca, Cca, Dca, Eca, Fca) alongside with additional criteria characterizing smoke production, occurrence of flaming droplets and acidity of thermal decomposition products. These tighter requirements have the purpose to protect people, property and environment by further reducing fire risk, smoke formation and delaying ignition of a material and fire outbreak.

These CPR regulations for HFFR (Halogen Free Flame Retardant) compounds can be fulfilled by using mineral flame retardants like aluminium hydroxide (ATH) or magnesium hydroxide (MDH). However, filling levels of 55-70 wt.-% (weight-%) of the metal hydrates need to be applied in order to comply with the demanding fire performance requirements. These high filling levels can impair the compound processability, furthermore the mechanical properties of the compound can be negatively influenced. Therefore, the additional incorporation of flame retardant synergists and process additives is frequently required. Known synergistic flame retardants like organically modified nanoclays, though, very often deteriorate compound processability and aging performance. Furthermore, the addition of an expensive stabilizer/antioxidant package may become necessary. With our new **ACTILOX® PA-B2** we are able to overcome these issues. This novel mineral based flame retardant synergist improves both processing and flame retardancy.

ACTILOX® PA-B2 is a white, powdery masterbatch containing metal hydrate and siloxane. It can be characterized by its high temperature stability and excellent polymer compatibility. Furthermore, it is easy to blend with all known mineral fillers. In basic compound formulations **ACTILOX® PA-B2** showed synergistic effects in combination with ATH resulting in an improved flame retardancy and hot air aging performance while providing a reduced pressure build-up during processing.

2. Improved processing behavior – ACTILOX® PA-B2 as processing aid

2.1. Compounding on a twin screw extruder

The positive influence of the new **ACTILOX® PA-B2** on the processing behavior was studied by compounding a simple PE/EVA formulation with 65 wt.-% filling level on a twin screw extruder. More exactly, the processability of a compound with 61 wt.-% **APYRAL® 40CD** and 4 wt.-% **ACTILOX® PA-B2** was studied. As reference a compound with solely **APYRAL® 40CD** (65 wt.-%) and a combination of **APYRAL® 40CD** (62 wt.-%) and a commercially available nanoclay (3 wt.-%) (NC), which is commonly applied as flame retardant synergist, was used. As the following table shows the addition of **ACTILOX® PA-B2** leads to the lowest mass pressure and mass temperature in the extruder of all three compounds.

Table 1. LLDPE/EVA formulations with corresponding compounding data.

Component [wt.-%]		65 wt.-% APYRAL® 40CD	61 wt.-% APYRAL® 40CD + 4 wt.-% ACTILOX® PA-B2	62 wt.-% APYRAL® 40CD + 3 wt.-% NC
LLDPE		13	13	13
EVA		17	17	17
PEgMA		5	5	5
APYRAL® 40CD		65	61	62
ACTILOX® PA-B2		-	4	-
Nanoclay (NC)		-	-	3
Characteristic compounding data				
Mass pressure	bar	75	68	84
Mass temperature	°C	175	170	185
Extruder usage	%	74	72	80
Comment				brittle, difficult to granulate

Additionally, the extruder usage was lower when **ACTILOX® PA-B2** was employed, 72% usage compared to 74% with **APYRAL® 40CD** and 80% with the **APYRAL® 40CD**/NC mixture. Thus, the overall energy consumption can be reduced by the application of **ACTILOX® PA-B2**.

2.2. Compounding in an internal kneader

In a next step, all three above mentioned formulations were compounded in an internal kneader and the resulting torque was recorded. As visible in figure 1, the compound with **ACTILOX® PA-B2** (red) shows the lowest torque while the compound with the nanoclay (green) shows the highest torque and a delayed melting of the compound, which translates into a prolonged processing time.

2.3. Filter pressure test

Using the so called filter pressure test (acc. to DIN EN 13900-5) the quality of the filler dispersion in the polymer matrix and the influence of mineral fillers on the pressure build-up during compounding and extrusion due to blocked screens can be determined. For this purpose granulated compound, which was obtained from twin screw extrusion, was melted in a single-screw-extruder and conveyed through a 50 µm filter. Finally, the pressure build-up in front of this filter was measured and evaluated.

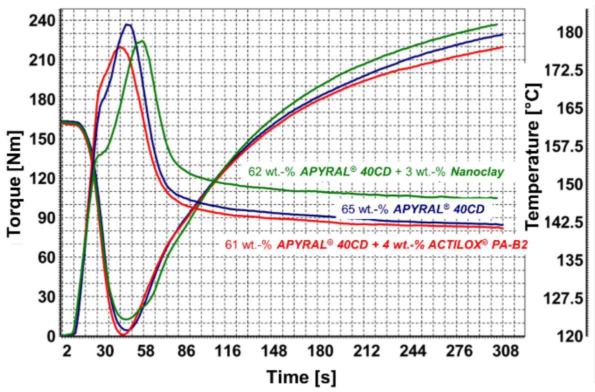

Figure 1. Torque and mass temperature during compounding of LLDPE/EVA formulations.

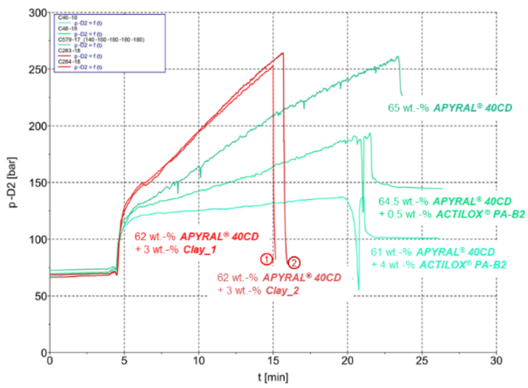

Figure 2. Pressure build-up over time of different LLDPE/EVA formulations.

As can be seen in figure 2, **APYRAL® 40CD** shows a moderately but constantly increasing pressure, while the compounds with **APYRAL® 40CD** and two different nanoclays lead to a faster pressure build-up which then, in both cases, results in an automatic termination of the measurement as the maximum allowed extru-der pressure was reached. As visible, the combination of **APYRAL® 40CD** with the new **ACTILOX® PA-B2** shows a very low pressure build-up which in the end means longer pro-duction times until a screen change is necessa-ry. With regard to **ACTILOX® PA-B2**, two different addition levels were tested. A first improvement of the pressure build-up can be achieved with only 0.5 wt.-% of **ACTILOX® PA-B2**, though, when 4 wt.-% **ACTILOX® PA-B2** are employed, not only an excellent processing behavior can be obtained but also flame retardancy can be significantly improved (see chapter 3).

Besides the graphical and visual evaluation, it is possible to retrieve significant parameters from the filter pressure test. The most important value is the filter pressure value (FPV), which is the difference of the maximum pressure and the starting pressure divided by the mass of filler. Besides the FPV the slope of the graphs gives also a good indication how fast or slow the pressure increases. As shown in the table below, the addition of **ACTILOX® PA-B2** leads to a significantly lower FPV and also the slope (1.7 bar/min) indicates that the pressure is increasing very slowly which in fact means that less screen changes will be necessary during processing.

Table 2. Parameters from filter pressure test.

	Unit	65 wt.-% APYRAL® 40CD	61 wt.-% APYRAL® 40CD + 4 wt.-% ACTILOX® PA-B2
Filter pressure value (FPV)	bar/g	0.28	0.10
m	bar/min	7.8	1.7
p max	bar	258	138
Extruder pressure	bar	414	330

3. Influence on flame propagation

Flame retardancy is in many cases evaluated with the UL 94 test which determines the flame spread in a certain burning direction. More commonly used is the vertical version of the test, the so called UL 94 V test (DIN IEC 60695-11-10). In this case, the test specimen is fixed in vertical direction and the flame is coming from the bottom in a 0° and 45°, respectively, angle. Depending on the flame performance, the material can be classified as V-0, with the highest flame retardancy, V-1 or V-2 with lower flame retardancy. Another frequently used tool to characterize the efficiency of flame retardants is the Cone Calorimeter (ISO 5660-1). For this test, the specimen is placed under the conical heater, it is irradiated and the evolving burnable gases are ignited by a spark. Roughly speaking, the heat generation during the burning process is determined via the consumption of oxygen. Furthermore, the amount of smoke is conti-nuously measured.

3.1. UL 94 V test

Besides the enhanced processing behavior, our new synergist **ACTILOX® PA-B2** significantly improves flame retardancy while maintaining the good mechanical properties of the compound.

When using aluminium hydroxide like our **APYRAL® 40CD** as flame retardant, an UL 94 V-0 rating at a thickness of 1.6 mm is not easily achieved. In this case, a flame retardant synergist needs to be added to fulfil the flame retardancy requirements. The addition of just 4 wt.-% of our **ACTILOX® PA-B2** to **APYRAL® 40CD** leads to a UL 94 V-0 classification with very low burning rates while, at the same time, all mechanical parameter are only slightly influenced. Alter-natively, an UL 94 V-0 rating can be achieved by either adding more **APYRAL® 40CD** (70 wt.-%) or by the addition of a nanoclay (3 wt.-%) which, however, in both cases will lead to a very poor processability of the compound (see MVR in table 3). Furthermore, the addition of the nanoclay leads to a significant reduction in whiteness (see figure 3).

As already known, flame retardancy is getting better with increasing BET surface area of metal hydrate. Therefore, an UL 94 V-0 rating can more easily be obtained with **APYRAL® 60CD**

Table 3. Properties of LLDPE/EVA formulations with **APYRAL® 40CD** and flame retardant synergists.

	Unit	65 wt.-% APYRAL® 40CD	70 wt.-% APYRAL® 40CD	61 wt.-% APYRAL® 40CD + 4 wt.-% ACTILOX® PA-B2	62 wt.-% APYRAL® 40CD + 3 wt.-% NC
Tensile strength	MPa	14.9	15.4	14.0	17.3
Elongation at break	%	150	112	168	154
MVR (190 °C/21.6 kg)	cm^3/10 min	3.6	0.9	2.8	0.9
LOI	% O_2	42	46	42	41
UL 94 V [1.6 mm]		Not classified	V-0	V-0	V-0
UL 94V – total burning time of 5 specimen	s	8/484	0/6	6/15	0/13
Shore D		58	60	55	59
Whiteness (Berger)		57.9	55.7	60.4	23.0

instead of **APYRAL® 40CD**. In this case, 59 wt.-% **APYRAL® 60CD** are sufficient to get the V-0 rating at 1.6 mm. Nevertheless, by addition of **ACTILOX® PA-B2**, the total filling level required to get an V-0 classification can be reduced from 59 wt.-% to 55 wt.-%. Furthermore, processability as well as mechanical properties can be clearly improved (see Table 4).

Figure 3. Addition of NC reduces whiteness.

3.2. Effects on Cone Calorimetry measurement

As mentioned before, the Cone Calorimetry measurement is used to further characterize flame retardancy. There are many parameters which can be retrieved from this measurement, for example the Heat Release Rate (HRR) which describes the amount of heat released over the analysis time. The Peak of the HRR (PHRR) describes the maximum value of the HRR graph. Starting from there, the Total Heat Release (THR) can be calculated which means the complete amount of heat which is released during the analysis time. As can be seen in the figure 4, the addition of **ACTILOX® PA-B2** in a LLDPE/EVA matrix leads to a significantly lower PHRR even though the THR is comparable.

Another interesting parameter which can be calculated is the Average Rate of Heat Emission (AHRE) as well as the Maximum of the AHRE (MAHRE). Again, the positive effect of **ACTILOX® PA-B2** is visible as the combination of **APYRAL® 40CD** with **ACTILOX® PA-B2** in a LLDPE/EVA matrix results in a lower AHRE and MAHRE (see figure 4).

Table 4. Properties of LLDPE/EVA formulations with **APYRAL® 60CD** and flame retardant synergists.

	Unit	59 wt.-% APYRAL® 60CD	57 wt.-% APYRAL® 60CD	52 wt.-% APYRAL® 60CD + 3 wt.-% ACTILOX® PA-B2	53 wt.-% APYRAL® 60CD + 2 wt.-% ACTILOX® PA-B2
Tensile strength	MPa	15.1	14.6	13.8	14.2
Elongation at break	%	196	266	383	395
MVR (190 °C/21.6 kg)	cm^3/10 min	3.6	5.3	6.4	6.7
LOI	% O_2	33	31	33	32
UL 94 V [1.6 mm]		V-0	Not classified	V-0	Not classified

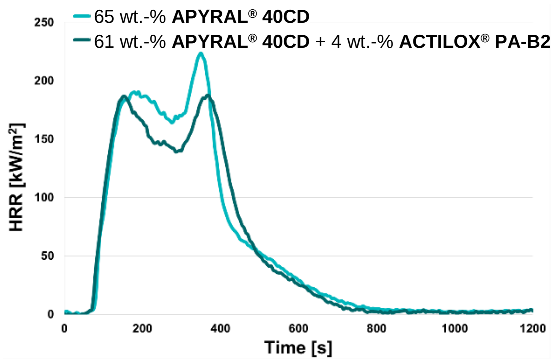

Figure 4. Heat Release Rate of LLDPE/EVA formulations.

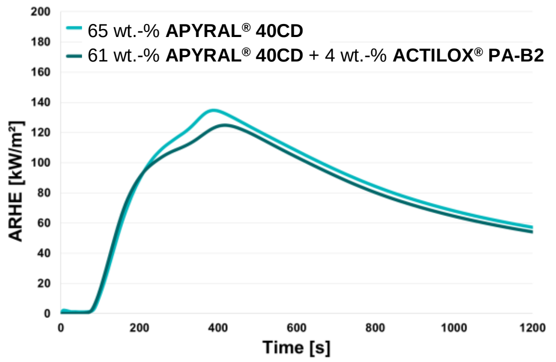

Figure 5. Average Rate of Heat Emission of LLDPE/EVA formulations.

4. Aging behavior of compounds

4.1. Hot Air Aging

For a cable compound the preservation of mechanical properties like tensile strength and elongation at break after aging is decisive. Therefore, the Hot Air Aging performance (DIN EN 60811-401) of a simple LLDPE/EVA cable compound formulation and the effect of the addition of **ACTILOX® PA-B2** was studied. As can be seen figure 6 and 7, tensile strength is increasing and elongation at break is decreasing in both formulations. However, the compound with **ACTILOX® PA-B2** shows well balanced aging properties and even after the aging process, good mechanical properties could still be maintained.

4.2. Water uptake

Especially for cable compounds a low water uptake is a critical factor in order to pass certain aging tests which are performed under water bath conditions. Therefore, the influence of the new **ACTILOX® PA-B2** on the water uptake of our basic LLDPE/EVA cable compound formulation was studied. Figure 8 shows that **APYRAL® 40CD** leads to very low water uptake

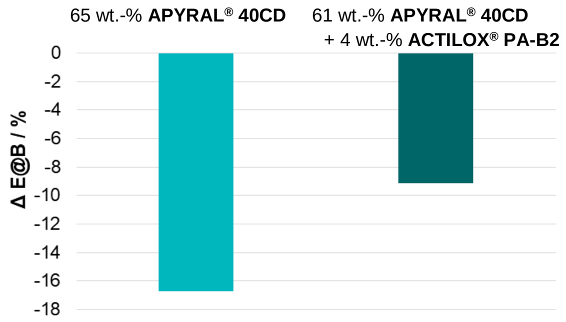

Figure 6. Change of elongation at break after Hot Air Aging.

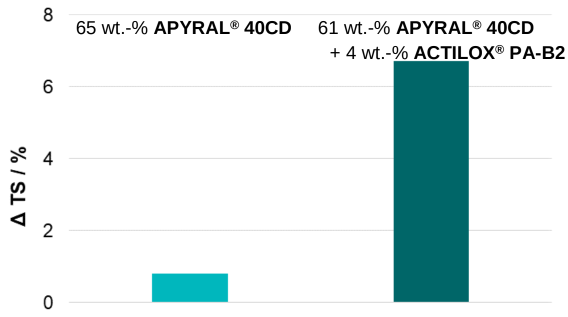

Figure 7. Change of tensile strength after Hot Air Aging.

of just 0.58% after 14 days at 70 °C. The addition of our ACTILOX® PA-B2 results in a slightly higher water uptake (0.77%) which, though, is still a comparable low value.

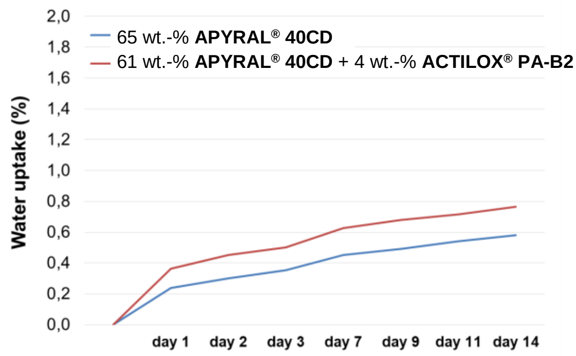

Figure 8. Water uptake at 70 °C for 14 days.

5. Application recommendation

In the end, the question remains what is the best dosage of **ACTILOX® PA-B2**. As always, the proper amount depends on the customers' specific formulation and needs to be tested. Nevertheless, in our investigations the best overall performance was obtained with a ratio of aluminium hydroxide (**APYRAL® 40CD**) and the synergist **ACTILOX® PA-B2** between 15:1 and

10:1 which means 4-6 wt.-% **ACTILOX® PA-B2** in a 65 wt.-% filling level. With regards to the combination of **APYRAL® 60CD** and **ACTILOX® PA-B2**, a ratio of 18:1 (3 wt.-% in 55 wt.-% filing level) is recommended. If the dosage of **ACTILOX® PA-B2** is too high, a deterioration of processing behavior can be observed. This was demonstrated by increasing by increasing the dosage of **ACTILOX® PA-B2** (0-12 wt.-%) in a LLDPE/EVA formulation with a total filling level of 65 wt.-%. It became visible that starting from an addition level of 8 wt.-%, an over-lubrication takes place which leads to a rippled surface (see figure 9). Furthermore, the flame retardancy performance is decreasing moderately when 8-12 wt.-% **ACTILOX® PA-B2** are applied are applied (see table 5).

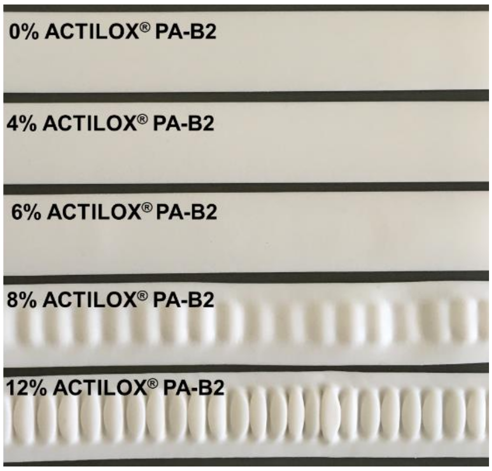

Figure 9. LLDPE/EVA formulation with varying **ACTILOX® PA-B2** addition levels.

Table 5. Properties of LLDPE/EVA formulations with varying **ACTILOX® PA-B2** addition levels.

	Unit	65 wt.-% APYRAL® 40CD	61 wt.-% APYRAL® 40CD + 4 wt.-% ACTILOX® PA-B2	59 wt.-% APYRAL® 40CD + 6 wt.-% ACTILOX® PA-B2	57 wt.-% APYRAL® 40CD + 8 wt.-% ACTILOX® PA-B2	53 wt.-% APYRAL® 40CD + 12 wt.-% ACTILOX® PA-B2
Tensile strength	MPa	14.4	12.6	11.5	11.5	10.7
Elongation at break	%	134	133	151	142	145
MVR (190 °C/ 21.6 kg)	cm^3/ 10 min	3.0	2.5	2.8	2.8	6.3
LOI	% O_2	40	43	45	42	42
UL 94 V		Failed	V-0	V-0	V-0	V-1