LAVIOSA

Dellite®

TECHNICAL HANDBOOK

Dellite®

25th

ANNIVERSARY

SUMMARY

OUR COMPANY

Founded in **1922**, Laviosa is a global leader in the **research, extraction, processing and distribution of bentonite and other clay-based products**. With a strong commitment to **sustainability**, the Group actively preserves and enhances the environment, ensuring responsible operations across all activities.

Our core strengths lie in the **control of raw materials, advanced process technologies**, and a **localized production footprint**, combined with **deep application expertise, efficient logistics**, and a **customer-centric approach.**

RESEARCH & DEVELOPMENT

R&D activities are structured within the internal **Laviosa Innovation HUB**, responsible for product development, process optimization, and technological advancement.

Continuous investment is dedicated to r**esearch and development programs** aimed at improving product performance in terms of quality, efficiency, and functional properties.

A significant portion of annual revenue is reinvested in innovation across processes, products, and services. This **systematic approach to innovation** enables the generation of proprietary technical know-how and strengthens Laviosa's positioning in the global market.

OUR PRESENCE

Laviosa Germany GmbH
Nordhorn (Germany)
PLANT

Laviosa France SAS
Ètréchy (France)
PLANT • SALES

Laviosa France SAS
Cersay (France)
MINES • PLANT

Laviosa Logistics France SAS
Veauche (France)
SALES

Laviosa Promasa SA
SUBSIDIARY
Vitoria (Spain)
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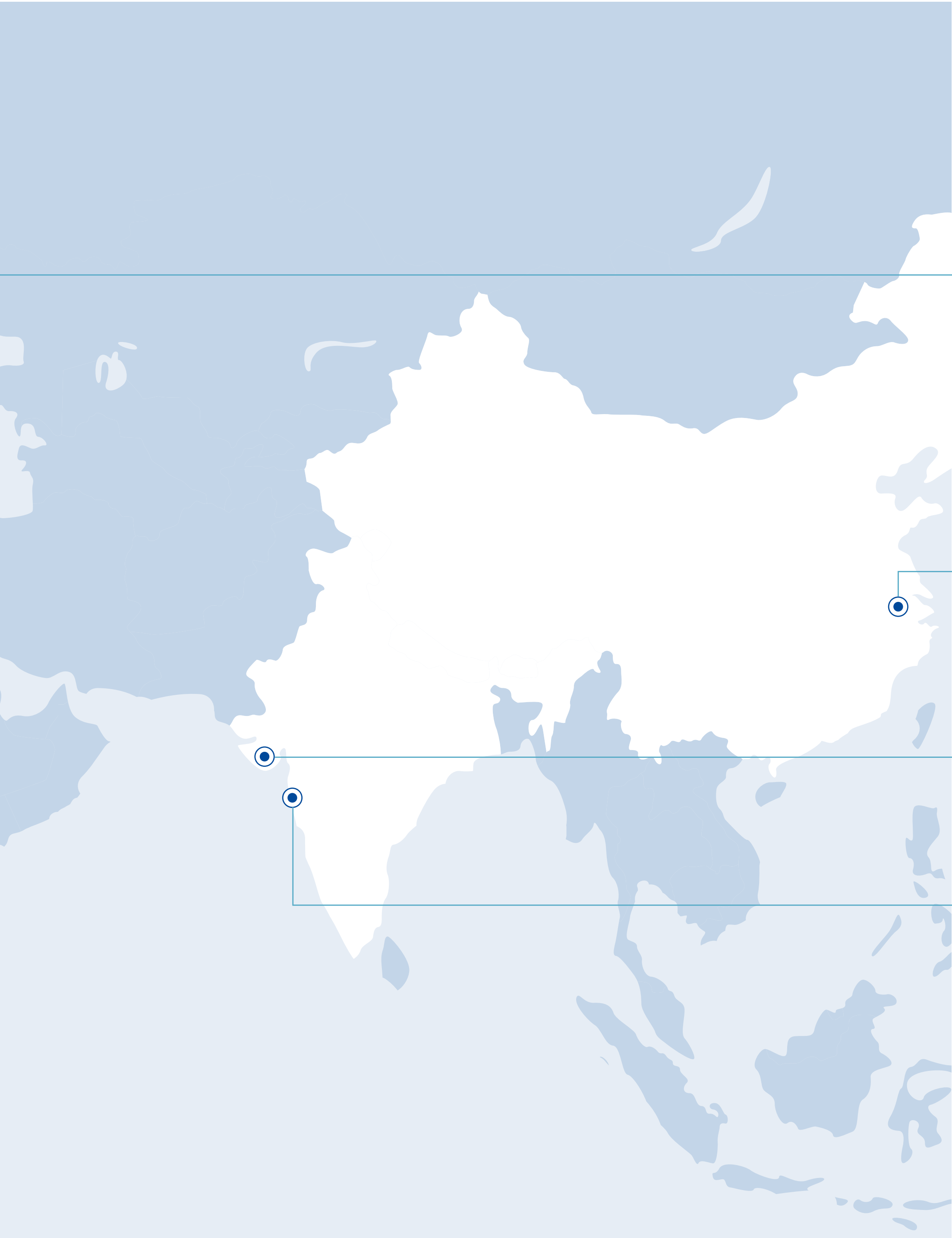
Laviosa Logistics Srl
Livorno (Italy)
HQ

Laviosa SpA
Livorno (Italy)
HQ • PLANT • SALES

Laviosa SpA
Villaspeciosa (Italy)
MINES • PLANT

IN THE WORLD



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Fatsa (Turkey)
SALES

Laviosa Sanayi Ve Ticaret Ltd Sti
Fatsa (Turkey)
MINES • PLANT • SALES

Zhejiang Hongyu New Materials Co Ltd
SUBSIDIARY
Si'an Town (China)
MINES • PLANT • SALES

Laviosa India Pvt Ltd
Pundi (India)
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Laviosa India Pvt Ltd
Mumbai (India)
SALES

Laviosa Logistics India Pvt Ltd
Mumbai (India)
SALES

Dellite®

25th ANNIVERSARY

Dellite® is an additive used in halogen-free flame-retardant cables and electrical wiring, which offers excellent performance in terms of thermal stability, blocking of toxic gases and anti-drip action, reducing the risk of fire.

Dellite® marks its 25th anniversary, celebrating a quarter century of innovation, research and development. This milestone reflects Laviosa's ability to transform the natural properties of minerals into advanced solutions for the future.

1. INTRODUCTION

The core of **Dellite®** is constituted by bentonite which is a swelling clay consisting mostly of montmorillonite (family of smectite) that is formed from volcanic ash, which converts the volcanic glass (obsidian) present in that ash into clay minerals.

It is though a natural origin material that doesn't present any contaminants in terms of PFAS, PFOS and other dangerous substances.

Dangerous contaminants concentration does not arise even after our controlled exchange reaction with quaternary ammonium salts to produce organically modified bentonite (organoclays).

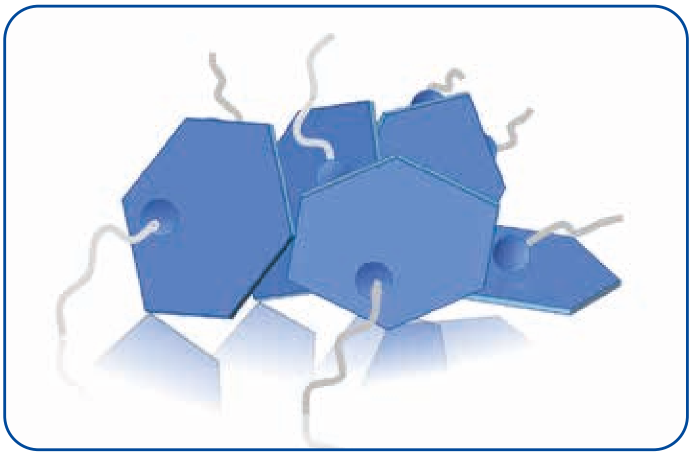

Figure 1: Schematic representation of organically modified bentonite platelets (organoclay).

The reaction allows Dellite® to be dispersed (expholiated) in polymer matrices to impart:

BARRIER PROPERTIES

INCREASED MECHANICAL PROPERTIES

BETTER FLAME RESISTANCE AND ANTIDRIPPING

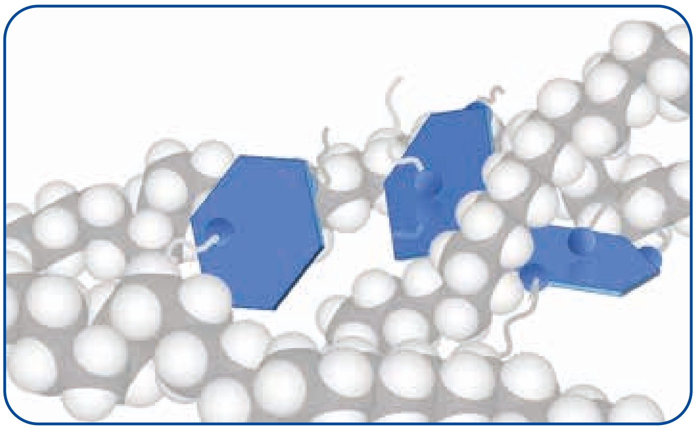

Figure 2: Schematic representation of exoliated Dellite® into a polymer matrix.

Barrier Properties

A proper dispersion of Dellite® platelets enhances barrier properties by forming a tortuous path for the gas molecules (like oxygen, water, CO2) permeating through the polymer matrix chains.

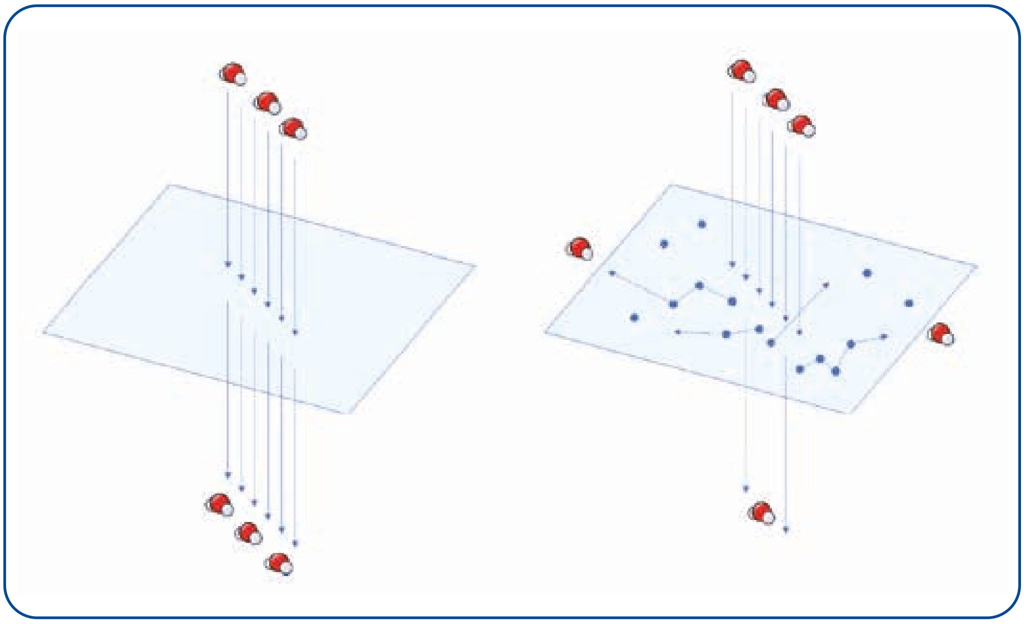

Figure 4: Dependence of gas permeability from the concentration of Dellite® in a polymer layer.

A preliminary study was carried out to understand the influence of the amount of Dellite® in these systems. Average data are reported in figure below.

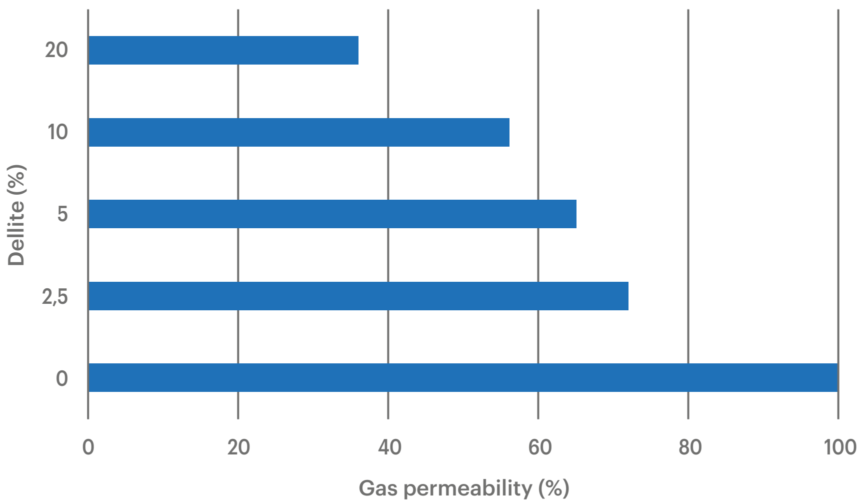

Figure 4: Dependence of gas permeability from the concentration of Dellite® in a polymer layer.

Mechanical Properties

The exfoliation on Dellite® into a polymer matrix reinforces the compound increasing the tensile strength, enhancing the overall elastic modulus.

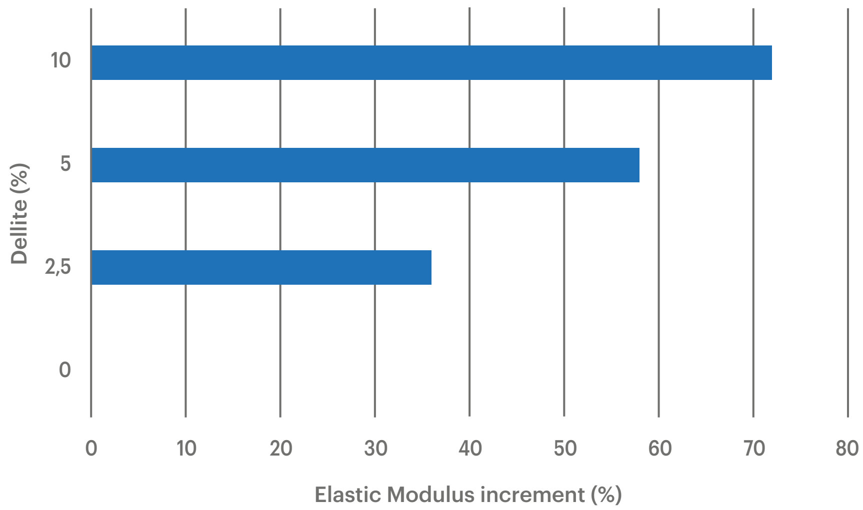

Figure 5: Elastic modulus trend with Dellite® concentration.

In figure X is reported the trend of elastic modulus with increasing amount of Dellite®.

Flame retardancy

Formation of a carbonaceous char is an effective method of increasing the fire resistance of materials. The material which forms char has a reduced ability to supply the flammable volatile compounds, required to fuel the fire. In addition to that, char acts as a physical barrier between the polymer and the superficial zone where the combustion of the polymer is occurring.

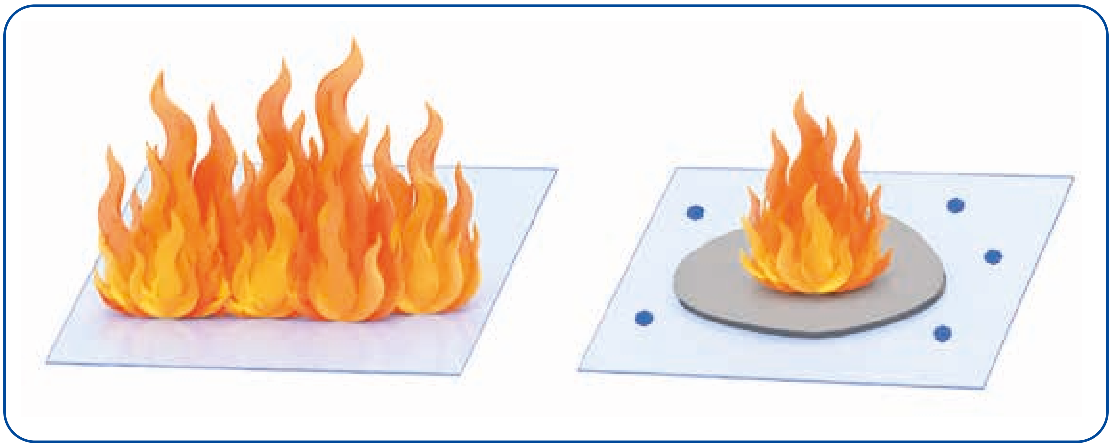

Figure 6: Schematic representation of char formation to avoid flame spreading.

Our products are synergistic with most flame-retardant systems such as heat absorbers, intumescent systems and radical cappers, promoting char formation and preventing the dripping of burning droplets.

2. PRODUCT PORTFOLIO

Here is our current portfolio:

PRODUCT	EVA	PE	PP	PA	TPE	PET/PBT	PLA
Dellite 72T	■	■	■	□	■	□	□
Dellite CW9	■	■	□	□	■	□	□
Dellite 43B	■	■	■	■	□	□	■
Dellite 67G	■	■	□	□	□	□	□
Dellite HPS	□	□	□	■	□	■	■
Dellite LVF	□	□	□	■	□	■	■
Dellite JFD	□	□	□	■	□	■	■
Dellite EFB	□	□	□	■	□	■	■

■ First recommendation

□ Second recommendation

3. DELLITE® IN EVA BASED HFFR COMPOUNDS

The first test was aimed at finding differences between Dellite® grades in common Halogen Free Flame Retardant (HFFR) formulations.
Alle the clays were dispersed and exfoliated in a twin hot roll mixer, so a modulable shear was applied.
Up to L06 organically modified clay are shown, fromL07 to L09 non modified clays.

Table 2: First tests in EVA based HFFR compound

EVA based HFFR compound	L01	L02	L03	L04	L05	L06
EVA28 MFI=3	23,5	23,5	23,5	23,5	23,5	23,5
mLLDPE MFI=3-5	8	8	8	8	8	8
LLDPE-g-MAH	4	4	4	4	4	4
fine pp ATH Martinal OL104LEO	45	45	45	45	45	45
$CaCO_3$ Microcarb SB	18	15	16	16	16	16
Silicon MB (Silmaprocess AL1142A)	1	1	1	1	1	1
Stabilizer AO (Silmastab AE1527E)	0,5	0,5	0,5	0,5	0,5	0,5
Dellite 72T		3	2			
Dellite 67G				2		
Dellite 43B					2	
Dellite CW9						2
Dellite LVF						
Dellite HPS						
Dellite JFD						

In this case, an EVA based blank formulation, employing aluminium tri-hydroxide (ATH) as main flame retardant, was used as reference. In subsequent formulations 3% or 2% of Dellite® were added.

Table 3: Mechanical properties before and after ageing of HFFR compound

Properties	L01	L02	L03	L04	L05	L06
Density at 23°C (g/cm^3)	1,543	1,53	1,536	1,534	1,538	1,534
MFI–21,6kg @ 190°C (g/10min)	9,6	5,8	6,8	10,4	5,8	9,7
Tensile strength (MPa)	12,7	12,5	11,8	11	12,1	10,7
Elongation at break (%)	162	160	154	160	151	175
ΔTS after 7dd@100°C in oven	4%	-4%	3%	2%	0%	6%
ΔE@B after 7dd@100°C in oven	-7%	-6%	-2%	5%	-6%	-18%
ΔTS after 7dd@70°C in water	-16%	-38%	-32%	-35%	-38%	-38%
ΔE@B after 7dd@70°C in water	-25%	-30%	-19%	36%	-32%	-13%
Water absorbtion	1,39%	3,86%	1,66%	2,60%	3,08%	2,56%
Initial Moisture Dellite (30'@120°C)		2,71	2,71	2,75	3,43	2,78

As it is clear from table 3 the amount of same Dellite® impacts the melt flow index (L02 and L03), the higher the amount the more detectable is the issue. In any case, not all Dellite® grades impacts the melt flow index in the same way. **It was found that the higher the amount of organic modifier, the more similar the melt flow is to the reference.**
Mechanical parameters are good in terms of Tensile Strength except for Dellite® CW9, which on the contrary has the highest Elongation at break.
The 100 °C ageing in air does not affect the mechanical parameters, but **in water only 72T shows a low water uptake after ageing, leading to an acceptable behaviour.**

Table 4: Fire behaviour of tested compounds

Vertical fire test (1.5 mm thickness)	L01	L02	L03	L04	L05	L06
Time 1. Top of the flame to high limit	106	153	193	80	190	90
Time 2. Flame base to graduation line	176	343	270	186	310	214
Self-extinguish	NO	NO	NO	NO	NO	NO
Presence of burning drops	YES	NO	NO	NO	NO	NO
Starting time of burning drops	24	-	-	-	-	-
Flame height	26	23	30	30	30	33
Flame width at graduation line	8	6	8	8	7	8

All the organic modified grades developed antidripping behaviour as expected. Dellite® 67G has the lowest impact on flame retardancy amongst the organic grades. Dellite® 72T and 43B performed the best.
To overcome the impact on mechanical properties after water ageing, a new formulation was developed.

Table 5: New formulation to avoid water ageing effects

EVA based HFFR compound	E00	E01	E02	E03
EVA28 MFI=3	22	22	22	22
mLLDPE MFI=3-5	12	12	12	12
LLDPE-g-MAH	4	4	4	4
fine pp ATH MartinalOL104LEO	60	58	58	58
Silicon MB (Silmaprocess AL1142A)	1,25	1,25	1,25	1,25
Stabilizer AO (Silmastab AE1527E)	0,25	0,25	0,25	0,25
Anti-hydrolysis MB (Silmastab AX2244)	0,5	0,5	0,5	0,5
Dellite 72T		2		
Dellite 67G			2	
Dellite CW9				2

As it is shown in table 5 calcium carbonate was depleted in favour of 60% of ATH and an anti-hydrolysis stabilizer was included.

Table 6: Mechanical properties before and after ageing of new HFFR compound

Properties	E00	E01	E02	E03
Density at 23°C (g/cm^3)	1,498	1,471	1,47	1,469
MFI-21,6kg @ 190°C (g/10min)	10,7	6,7	10,7	6
Tensile strenght (Mpa)	13,7	11,7	11	11,2
Elongation at break (%)	163	141	143	189
TS at 100% (Mpa)	12,8	11,7	11	11,1
ΔTS after 7dd@100°C in oven	-8%	21%	13%	12%
ΔE@B after 7dd@100°C in oven	-14%	15%	10%	-21%
ΔTS after 7dd@70°C in water	-5%	-6%	-2%	0%
ΔE@B after 7dd@70°C in water	2%	15%	4%	-11%
Water absorbtion	0,36%	0,79%	1,21%	1,22%

As it possible to observe in table 6, the **addition of the hydrolysis stabilizer, completely avoided water ageing effect due to the presence of modified clays.**

Table 7: Fire behaviour of tested compounds

Vertical fire test (1.5 mm thickness)	E00	E01	E02	E03
Time 1. Top of the flame to high limit	100	153	128	147
Time 2. Flame base to graduation line	210	302	226	331
Self extinguish	NO	NO	NO	NO
Presence of burning drops	YES	NO	YES	NO
Starting time of burning drops	49		207	
Flame height	27	28	32	29
Flame width at graduation line	7	8,5	9	6

In table 7 an anomalous behaviour of Dellite® 67G was observed, no droplets but very fluid melted polymer, while 72T and CW9 showed similar positive effects on flame retardancy, with slightly better behaviour of CW9. For this reason, the reproducibility of results was evaluated for Dellite® 72T and Dellite® CW9.
Each grade of Dellite® was tested in three different batches produced in Livorno, Organoclay Plant.

Table 8: Final formulations for reproducibility evaluation

EVA based HFFR compound	72T-1	72T-2	72T-3	CW9-1	CW9-2	CW9-3
EVA28 MFI=3	22	22	22	22	22	22
mLLDPE MFI=3-5	12	12	12	12	12	12
LLDPE-g-MAH	4	4	4	4	4	4
fine pp ATH MartinalOL104LEO	58	58	58	58	58	58
Silicon MB (Silmaprocess AL1142A)	1,25	1,25	1,25	1,25	1,25	1,25
Stabilizer AO (Silmastab AE1527E)	0,25	0,25	0,25	0,25	0,25	0,25
Anti-hydrolisis MB (Silmastab AX2244)	0,5	0,5	0,5	0,5	0,5	0,5
Dellite 72T (1° sample)	2					
Dellite 72T 05110		2				
Dellite 72T 19040			2			
Dellite CW9 (1° sample)				2		
Dellite CW9 11100					2	
Dellite CW9 13051						2

Mechanical properties are homogeneous, with acceptable standard deviation within same products as shown below.

Table 9: Mechanical properties before and after ageing of HFFR compound for reproducibility evaluation

Properties	72T-1	72T-2	72T-3	CW9-1	CW9-2	CW9-3
Density at 23°C (g/cm^3)	1,471	1,476	1,479	1,469	1,471	1,466
MFI-21,6kg @ 190°C (g/10min)	6,7	5,4	7,8	6	4	7
Tensile strenght (Mpa)	11,7	12,7	12,4	11,2	12,3	10,9
Elongation at break (%)	141	140	144	189	195	164
TS at 100% (Mpa)	11,7	12,4	12,1	11,1	12	10,9

Table 10: Fire behaviour of tested compounds

Vertical fire test	72T-1	72T-2	72T-3	CW9-1	CW9-2	CW9-3
Time 1. Top of the flame to high limit	153	179	144	147		
Time 2. Flame base to graduation line	302	270	280	331		
Self-extinguish	NO	NO	NO	NO	YES	YES
Self-extinguish time					200	270
Presence of burning drops	NO	NO	NO	NO	NO	NO
Flame height	28	25	26	29	13	13
Flame width at graduation line	8,5	6,5	6	6	3	3

As it is clear from table 10 all the tested Dellite® samples showed a good flame retardancy enhancement, but 2 out of 3 samples of compounds containing **Dellite® CW9 reached self-extinguishing grade.**

Conclusion

In conclusion, in EVA/ATH based HFFR compounds organophilic bentonites are very suitable for this kind of matrix with some distinctions:

- **Dellite® 43B** – good behaviour with no particular recommendation.
- **Dellite® 67G** – develops quite good synergistic behaviour with ATH, its key point is the very low impact on MFI.
- **Dellite® 72T** – one of the best in terms of flame retardancy, its key point are the good reinforcement that gives to the compound and the very low water uptake that allow a retention of mechanical properties after water ageing.
- **Dellite® CW9** – the best in flame retardancy (reaches even self-extinguishing in this kind of recipes) and elongation is generally the highest. It works better with an anti-hydrolysis additive.

4. DELLITE® IN POE BASED HFFR COMPOUNDS

Another screening was focused on studying the behaviour of Dellite® in elastomeric polyolefins (POE) and their synergy with magnesium di-hydroxide (MDH).

Table 11: First tests in POE/MDH based HFFR compound

POE based HFFR compound	P00	P01	P02	P03	P04
Engage 8003	24	24	24	24	24
mLLDPE MFI=3-5	10	10	10	10	10
LLDPE-g-MAH	3,5	3,5	3,5	3,5	3,5
Ecopiren 3.5	60	58	58	58	58
Silicon MB (Silmaprocess AL1142A)	1,5	1,5	1,5	1,5	1,5
Stabilizer AO (Silmastab AE1527E)	0,5	0,5	0,5	0,5	0,5
Anti-hydrolisis MB (Silmastab AX2244)	0,5	0,5	0,5	0,5	0,5
Dellite 72T		2			
Dellite 67G			2		
Dellite CW9				2	
Dellite 43B					2

Based on knowledge from EVA based compound, the addition of anti-hydrolysis additive was kept, in order to overcome water ageing.

Table 12: Mechanical properties before and after ageing of HFFR compounds

Properties	P00	P01	P02	P03	P04
Density at 23°C (g/cm^3)	1,445	1,434	1,428	1,43	1,432
MFI-21,6kg @ 190°C (g/10min)	4,2	4,7	4,3	3,6	3,5
Tensile strenght (MPa)	15,7	13,7	12,6	12,4	13,4
Elongation at break (%)	148	153	149	173	158
TS at 100% (Mpa)	15,6	13,7	12,7	12,5	13,5
ΔTS after 7dd@100°C in oven	-11%	5%	9%	10%	4%
ΔE@B after 7dd@100°C in oven	-6%	7%	4%	-8%	-10%
ΔTS after 7dd@70°C in water	-32%	-21%	-18%	-19%	-25%
ΔE@B after 7dd@70°C in water	32%	21%	112%	36%	45%
Water absorbtion	0,7%	0,8%	1,0%	0,9%	1,2%

The addition of the **anti-hydrolysis stabilizer wasn't enough to avoid differences below 30% for all the compounds, except for Dellite® 72T** that showed the best retention of mechanical properties due to the lowest amount of water absorbed. This failure was probably due to the presence of brucite, a **natural occurring MDH powder with high hygroscopicity.**

Table 13: Fire behaviour of tested compounds

Vertical fire test	P00	P01	P02	P03	P04
Time 1. Top of the flame to high limit	78	57	68	100	74
Time 2. Flame base to graduation line	160	215	206	242	220
Self extinguish	NO	NO	NO	NO	NO
Presence of burning drops	YES	NO	NO	NO	NO
Starting time of burning drops	141				
Flame height	32	34	34	28	32
Flame width at graduation line	6	9,5	9	8	8

Again, as it is possible to understand from table 13, the behaviour is similar in terms of **antidripping** with Dellite® CW9.

Table 14: POE based formulations for reproducibility evaluation

POE based HFFR compound	72T-1	72T-2	72T-3	CW9-1	CW9-2	CW9-3
Engage 8003	24	24	24	24	24	24
mLLDPE MFI=3-5	10	10	10	10	10	10
LLDPE-g-MAH	3,5	3,5	3,5	3,5	3,5	3,5
Ecopiren 3.5	58	58	58	58	58	58
Silicon MB (Silmaprocess AL1142A)	1,5	1,5	1,5	1,5	1,5	1,5
Stabilizer AO (Silmastab AE1527E)	0,5	0,5	0,5	0,5	0,5	0,5
Anti-hydrolisis MB (Silmastab AX2244)	0,5	0,5	0,5	0,5	0,5	0,5
Dellite 72T (1° sample)	2					
Dellite 72T 05110		2				
Dellite 72T 19040			2			
Dellite CW9 (1° sample)				2		
Dellite CW9 11100					2	
Dellite CW9 13051						2

Results are shown below. Lower homogeneity can be observed, compared to EVA/ATH based compounds.

Table 15: Mechanical properties before and after ageing of HFFR compound for reproducibility evaluation

Properties	72T-1	72T-2	72T-3	CW9-1	CW9-2	CW9-3
Density at 23°C (g/cm^3)	1,434	1,429	1,427	1,43	1,438	1,433
MFI–21,6kg @ 190°C (g/10min)	4,7	4,3	3	3,6	3,2	3,8
Tensile strength (MPa)	13,7	12,6	11,7	12,4	11,8	11,4
Elongation at break (%)	153	180	155	173	196	155

Table 16: Fire behaviour of tested compounds

Vertical fire test	72T-1	72T-2	72T-3	CW9-1	CW9-2	CW9-3
Time 1. Top of the flame to high limit	57	75	88	100	90	122
Time 2. Flame base to graduation line	215	230	248	242	254	245
Self-extinguish	NO	NO	NO	NO	NO	NO
Presence of burning drops	NO	NO	NO	NO	NO	NO
Flame height	34	33	31	28	31	30
Flame width at graduation line	9,5	7,5	7,5	8	7	6,5

In this kind of recipe, no self-extinguishing can be observed, for any grade, but the trend is retained for all the batches with **Dellite® CW9 showing slightly better flame behaviour.**

Conclusion

In conclusion, in POE/MDH based HFFR compound no remarkable differences between Dellite® grades were observed. There is a synergic behaviour with natural MDH (brucite) in terms of flame retardancy, but its presence nullifies the effect of anti-hydrolysis stabilizer. For this reason, only Dellite® 72T retains mechanical properties after water ageing. So, been the flame retardancy comparable, with mechanical properties slightly better than the other grades, **Dellite® 72T is the most suitable for this system.**

5. COMPARISON BETWEEN DELLITE® GRADES AND SOME COMPETITORS IN EVA/ATH BASED HFFR COMPOUNDS

In the study below, it is presented a comparison between different organoclays, added at 2% on HFFR EVA based and POE based compounds with 60% flame retardant (ATH or MDH).
Competitors' grades were evaluated to understand their general information and, if any, some similarities with our products.

Table 17: General characterizations of nanoclays for plastics

	Sympatec Analisys			Residue		Gravimetrics	
Product	D10 (μm)	D50 (μm)	D90 (μm)	45 μm	75 μm	U%	LOI%
DELLITE CW9	7,6	27,3	51,6	8,6	1,5	1,5	46,1
DELLITE 72T	3,8	18,4	42,3	2,7	0,1	1,8	38,2
DELLITE 67G	6,2	24,3	50,7	5,2	0,1	1,4	43,9
Competitor 1-1	1,7	8,0	22,0	0,5	0,4	2,7	38,8
Competitor 1-2	2,9	9,7	26,9	32,4	31,2	1,1	56,8
Competitor 1-3	3,3	15,6	37,0	1,4	0,4	2,1	38,3
Competitor 2-1	2,9	12,7	29,6	50,8	0,9	3,9	28,0
Competitor 2-2	2,6	11,0	28,2	5,6	0,1	7,1	20,9

The products are very different in terms of loss on ignition (LOI) or granulometric profile, but there is a main similarity between **Dellite® 72T and Competitor 1-3. Their profile, residue and LOI are perfectly matching.**

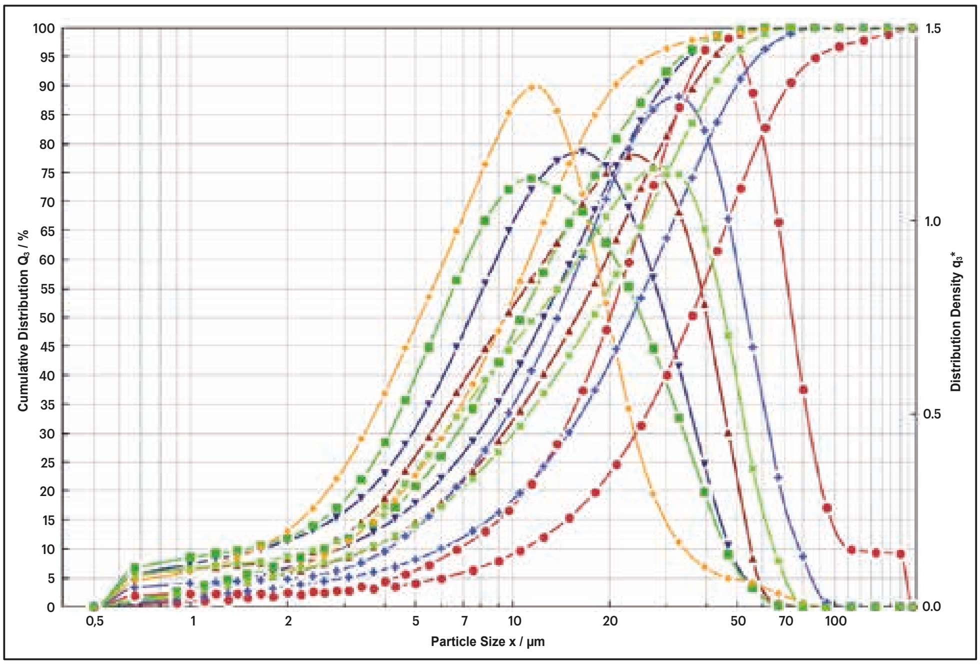

Figure 7: Sympatec analysis of all organoclay for plastic market

This similarity is confirmed also in figure 7 where **Competitor 1-3 and Dellite® 72T are overlapping.**

The base formulation is the one with anti-hydrolysis additive, ATH is used as reference and 2% of different clay was added and tested. It is a starting point for comparison tests.

Table 18: EVA/ATH based formulations with different organoclays

EVA based HFFR compound	ATH	Dellite 72T	Dellite CW9	Competitor 2-2	Competitor 1-3
EVA28 MFI=3	22	22	22	22	22
mLLDPE MFI=3-5	12	12	12	12	12
LLDPE-g-MAH	4	4	4	4	4
fine pp ATH MartinalOL104LEO	60	58	58	58	58
Silicon MB (Silmaprocess AL1142A)	1,25	1,25	1,25	1,25	1,25
Stabilizer AO (Silmastab AE1527E)	0,25	0,25	0,25	0,25	0,25
Anti-hydrolisis MB (Silmastab AX2244)	0,5	0,5	0,5	0,5	0,5
Dellite 72T		2			
Dellite CW9			2		
Competitor 2-2				2	
Competitor 1-3					2

As it is clear from table 19, Competitor 1-3 has a very negative impact on MFI, leaving the material nearly unworkable. It also shows the highest reinforcement. Dellite® CW9 shows the best elongation at break.

Table 19: Mechanical properties of different nanocomposites

Properties	ATH	Dellite 72T	Dellite CW9	Competitor 2-2	Competitor 1-3
Density at 23°C (g/cm³)	1,498	1,475	1,469	1,476	1,476
MFI-21,6kg @ 190°C (g/10min)	10,7	6,6	5,7	8,7	1,6
Tensile strenght (Mpa)	13,7	12,3	11,5	12,1	12,7
Elongation at break (%)	163	142	183	161	146

Table 20: Flame behaviour of tested compounds

Vertical fire test	ATH	Dellite 72T	Dellite CW9	Competitor 2-2	Competitor 1-3
Time 1. Top of the flame to high limit	100	159		115	160
Time 2. Flame base to graduation line	210	284		335	332
Self-extinguish	NO	NO	YES	NO	NO
Presence of burning drops	YES	NO	NO	NO	NO
Starting time of burning drops	49				
Flame height	27	26	18	23	27
Flame width at graduation line	7	7	4	6	6

As presented in table 20, Dellite® 72T and Competitor 1-3 has a very similar synergy with ATH, while **Dellite® CW9 shows again self-extinguishing properties.**

The organoclays were also tested in POE/MDH based HFFR to compare their properties.

Table 21: POE/MDH based formulation with different organoclays

POE based HFFR compound	MDH	Dellite 72T	Dellite CW9	Competitor 2-2	Competitor 1-3
Engage 8003	24	24	24	24	24
mLLDPE MFI=3-5	10	10	10	10	10
LLDPE-g-MAH	3,5	3,5	3,5	3,5	3,5
Ecopiren 3.5	60	58	58	58	58
Silicon MB (Silmaprocess AL1142A)	1,5	1,5	1,5	1,5	1,5
Stabilizer AO (Silmastab AE1527E)	0,5	0,5	0,5	0,5	0,5
Anti-hydrolisis MB (Silmastab AX2244)	0,5	0,5	0,5	0,5	0,5
Dellite 72T		2			
Dellite CW9			2		
Competitor 2-2				2	
Competitor 1-3					2

The compounds in table 21 were evaluated as per standard method applied in previous studies.

Table 22: Mechanical properties of different nanocomposites

Properties	MDH	Dellite 72T	Dellite CW9	Competitor 2-2	Competitor 1-3
Density at 23°C (g/cm3)	1,445	1,430	1,434	1,431	1,399
MFI-21,6kg @ 190°C (g/10min)	4,2	4	3,5	3,1	2,4
Tensile strenght (Mpa)	15,7	12,7	11,9	14,9	11,9
Elongation at break (%)	148	163	175	173	130

As expected, being produced with sepiolite (at least a portion of it), the **reinforcement given by Competitor 2-2 is the highest amongst all samples without any ageing.**
Dellite® CW9 has the best elongation, as per previous tests and, 72T does not affect MFI keeping it at reference level.

Table 23: Flame behaviour of tested compounds

Vertical fire test	MDH	Dellite 72T	Dellite CW9	Competitor 2-2	Competitor 1-3
Time 1. Top of the flame to high limit	78	73	104	89	78
Time 2. Flame base to graduation line	160	231	247	290	268
Self-extinguish	NO	NO	NO	NO	NO
Presence of burning drops	YES	NO	NO	NO	NO
Starting time of burning drops	141				
Flame height	32	33	30	27	27
Flame width at graduation line	6	8	7	7	6,5

With this kind of recipe **no differences are evidenced, all the samples behave similarly.**

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To enhance the differences between Dellite® grades and competitors, other EVA/ATH based compounds were tested, adding the ageing effect at higher temperatures for the air ageing (130 °C instead of 100 °C) and taking out of recipe the stabilizer for water ageing.
A new organoclay was added to the rack, the Competitor 3-1, launched in 2024 on the market.

Table 24: EVA/ATH based formulations with different organoclays

EVA based HFFR compound	HF L-0	HF L-CW9	HF L-72T	HF L-1	HF L-2	HF L-3
EVA28 MFI=3 (ELVAX 265)	24	24	24	24	24	24
mLLDPE MFI=3-5 (EXCEED 3518)	10	10	10	10	10	10
LLDPE-g-MAH (FUSABOND E226)	4	4	4	4	4	4
fine pp ATH (MartinalOL104LEO)	60	58	58	58	58	58
Silicon MB (AL1142A plus)	1,5	1,5	1,5	1,5	1,5	1,5
Stabilizer (AE1527E)	0,5	0,5	0,5	0,5	0,5	0,5
Dellite CW9		2				
Dellite 72T			2			
Competitor 2-2				2		
Competitor 1-3					2	
Competitor 3-1						2

Table 25: Mechanical properties of different nanocomposites

Properties	HF L-0	HF L-CW9	HF L-72T	HF L-1	HF L-2	HF L-3
Density at 23°C (g/cm^3)	1,45	1,454	1,456	1,453	1,457	1,46
MFI-21,6kg @160°C (g/10 min)	9	8,6	6,2	5,6	6	6
Tensile strength (MPa)	14,9 ± 0,1	14,8 ± 0,2	14,9 ± 0,3	15,9 ± 0,4	14,9 ± 0,2	14,6 ± 0,7
Elongation at break (%)	213 ± 9	195 ± 22	186 ± 12	197 ± 10	193 ± 10	164 ± 14
Tensile strength at 100% Elong. (MPa)	13,0 ± 0,2	12,8 ± 0,1	13,1 ± 0,3	14,4 ± 0,3	12,9 ± 0,1	13,0 ± 0,5
Tensile strength at 200% Elong. (MPa)	15,1 ± 0,1	14,9 ± 0,1	15,0 ± 0,1	16,3 ± 0,3	15,1 ± 0,2	-

As it is clear from table 25, **adding 2% Dellite® CW9 does not change the MFI** compared to the reference, while adding the other organoclays (OCs) causes a lowering of the MFI compared to the reference.
Moving to mechanical properties, table 25 shows that the addition of organoclays does not change the tensile strength, except for Competitor 2-2 which causes an increase in the resistance. Regarding the effect on the elongation at break, it is possible to understand that the addition of Competitor 3-1causes a significant damping; the other organoclays do not change this parameter compared to the reference.

Table 26: Flame behaviour of tested compounds

Vertical burning test	HF L-0	HF L-CW9	HF L-72T	HF L-1	HF L-2	HF L-3
Time 1. Top of the flame to high limit (s)	67	103	106	80	85	111
Time 2. Flame base to graduation line (s)	171	244	256	210	281	269
Self extinguish (YES/NO)	NO	NO	NO	NO	NO	NO
Presence of burning drops (YES/NO)	YES	NO	NO	NO	NO	NO
Starting time of burning drops (s)	32	–	–	–	–	–
Flame height (cm)	>20	18	17-18	20	18	18-19
Flame width at graduation line (cm)	8-9	7	6-7	9	7	6

During the burning test, reference shows burned dripping after approximately 30 seconds of flame application, as well as fast and wide flame propagation all over the sample. All clays act as char formers and anti-dripping agents, preventing sample dripping by forming protective char on the surface, while also slowing down the flame propagation.

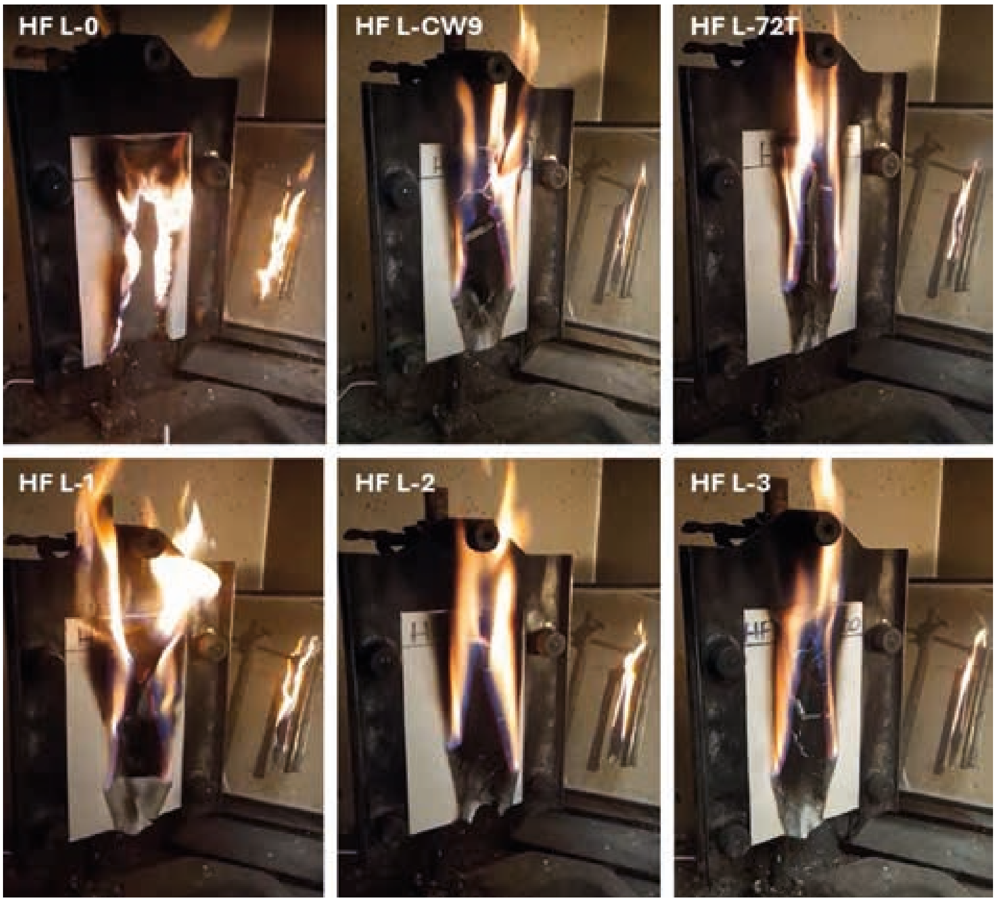

Figure 8: Vertical burning tests of different nanocomposites

The focus of this study was to understand the mechanical properties after water ageing and thermal ageing in air.

Table 27: Mechanical results after water ageing of different nanocomposites produced with different organoclays

Water Ageing [168h @70°C in water]	HF L-0	HF L-CW9	HF L-72T	HF L-1	HF L-2	HF L-3
Tensile strength (MPa)	11,9 ± 0,1	9,2 ± 0,1	11,0 ± 0,6	9,6 ± 0,3	10,6 ± 0,2	10,1 ± 0,4
Elongation at break (%)	176 ± 28	217 ± 23	175 ± 21	211 ± 36	200 ± 19	131 ± 6
Tensile strength at 100% Elongation (MPa)	12,7 ± 0,1	9,8 ± 0,2	11,3 ± 0,5	10,4 ± 0,3	10,9 ± 0,4	10,4 ± 0,3
Tensile strength at 200% Elongation (MPa)	–	9,3 ± 0,5	–	10,0 ± 0,3	10,8 ± 0,3	–
TS difference (%)	-20	-38	-26	-40	-29	-31
E@b difference (%)	-17	11	-6	7	4	-20
Water Uptake (%)	1	2,7	1,8	3,9	2	1,8

Table 28: Mechanical results after thermal ageing in air of different nanocomposites produced with different organoclays

Thermal ageing [168h @135°C in air]	HF L-0	HF L-CW9	HF L-72T	HF L-1	HF L-2	HF L-3
Tensile strength (MPa)	15,9 ± 0,2	14,6 ± 0,4	14,7 ± 0,5	16,4 ± 0,1	14,3 ± 0,7	13,6 ± 0,2
Elongation at break (%)	164 ± 17	117 ± 8	113 ± 16	127 ± 6	110 ± 19	102 ± 8
Tensile strength at 100% Elongation (MPa)	15,5 ± 0,1	14,4 ± 0,2	14,6 ± 0,4	17,1 ± 0,1	14,7 ± 0,1	13,5 ± 0,2
TS difference (%)	6,7	-1,4	-1,3	3	-4	-6,8
E@b difference (%)	-23	-40	-39	-36	-43	-38

By observing the differences between the pre and post water aging specimens, it is possible to observe that the reference and **Dellite® 72T perform at best in terms of tensile strength**; the reduction of the load is acceptable. Dellite CW9 and Competitor 2-2, on the other hand, show more strength loss than the pre-aging specimens, with a significant difference of 40%. The other samples show intermediate behavior. This behavior is related to water uptake, as both the reference sample and **Dellite 72T exhibit lower water absorption than the other organoclays.** Moreover, neither the reference sample nor the clay-containing compounds show swelling after water ageing.

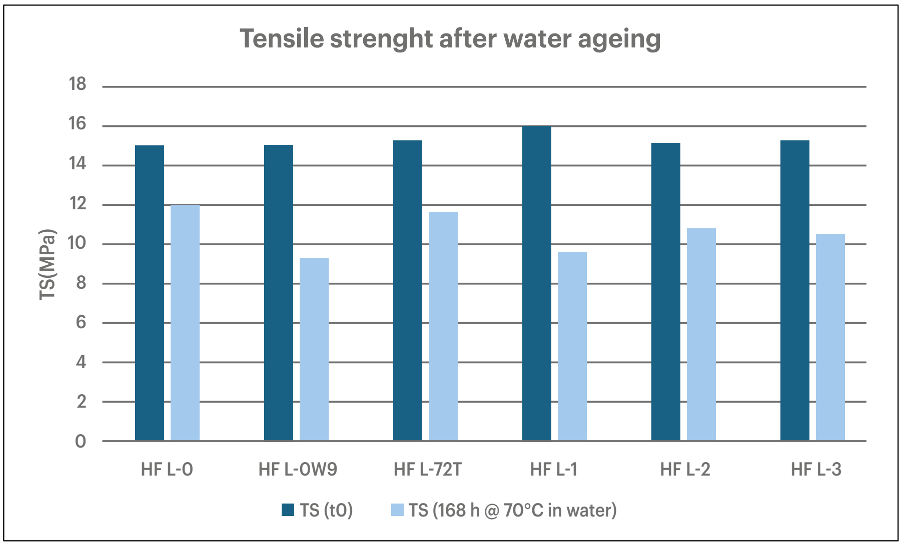

Figure 9: Mechanical properties after water ageing

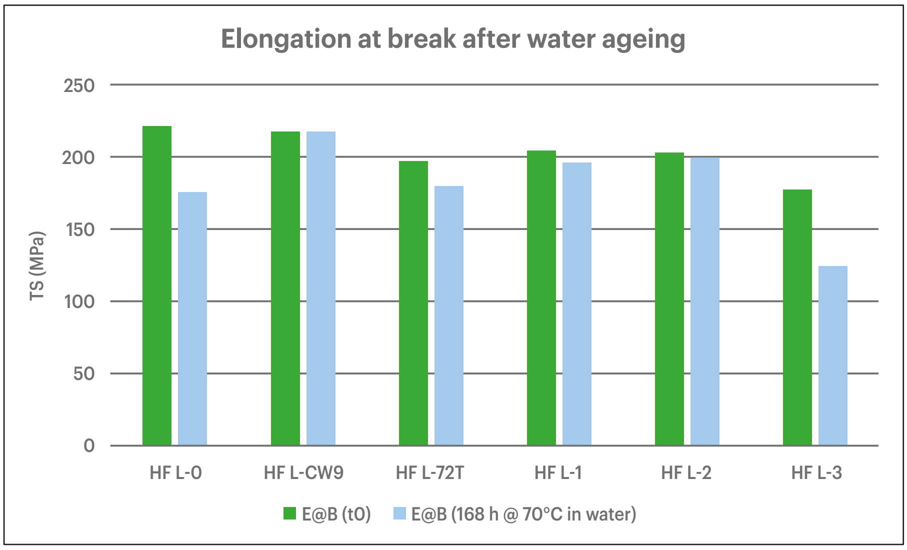

Figure 10: Elongation at break after water ageing

Looking at thermal ageing in air, except for the reference, which showed a difference in elongation at break of approximately 20% (acceptable), **all compounds show approximately 40% loss in breaking elongation post-aging.**

Even the color difference seems more marked for the samples with organoclays as it is shown in picture 11 and 12. Especially Competitor 1-3 and Competitor 3-1 are affected by this change in color.

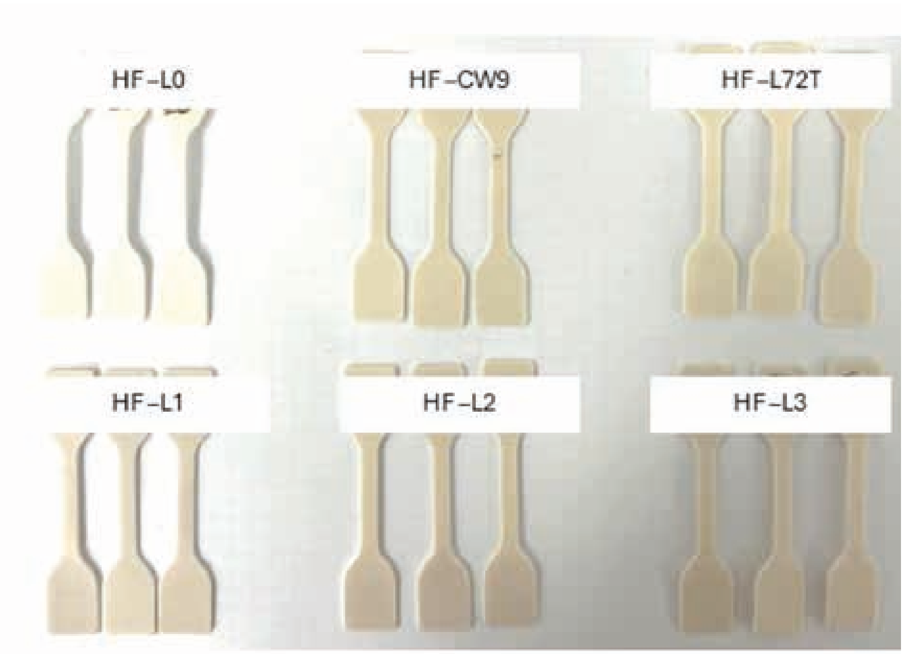

Figure 11: samples before thermal ageing in air

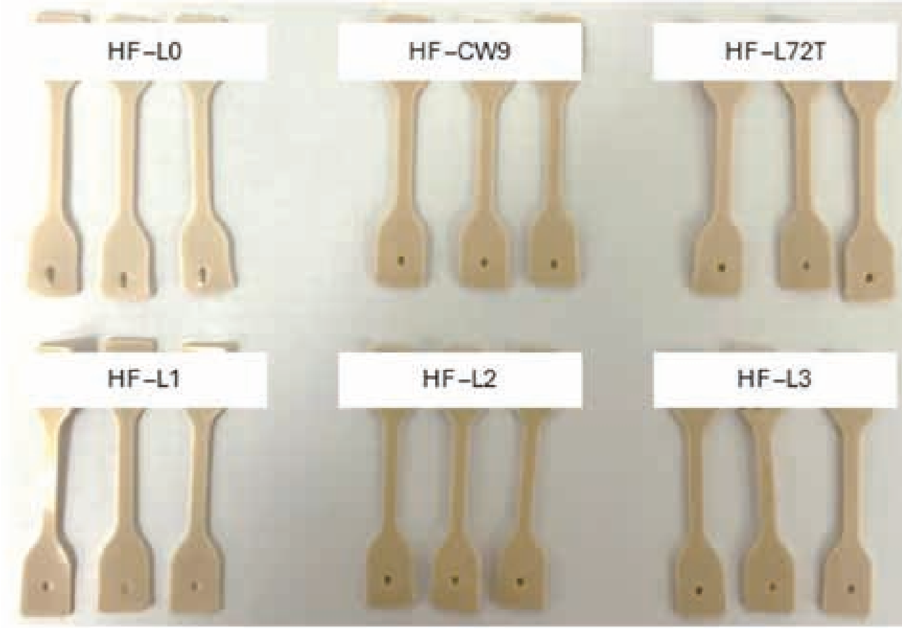

Figure 12: samples after thermal ageing in air

Conclusion

In conclusion, **Dellite® CW9 does not cause significant changes in the compound's MF**I, which remains substantially aligned with reference, retaining very good workability. However, thanks to the formation of a surface char, it can effectively eliminate the dripping, achieving a very positive result in this test. On the other hand, it shows a greater tendency to absorb water and moisture than both the reference and other commercial clays. Consequently, the compound containing 2% CW9 shows a damping in properties after aging in water.

On the other hand, **Dellite® 72T** induces a reduction in the compound's MFI compared to the reference but it proves as very effective in char forming and anti-dripping agent with a very positive result. Furthermore, it **exhibits very low water and moisture absorption**, substantially comparable to that of the reference; this translates into **better retention of mechanical properties after aging** in water, both compared to CW9 and other commercial clays.

6. STUDY ON AGEING BEHAVIOUR OF DELLITE®

Following the results obtained during air aging (1 week at 135°C) of the EVA based formulations with 2% Dellite® CW9 and 72T, an aging study was conducted employing Dellite® CW9 and modifying various components of the recipe to understand which component of the formulation, interacting with the QUAT, could cause problems.

Table 29: EVA/ATH based formulations for ageing studies

Eva based HFFR compound	HF L-0	HF L-CW9	HF L-coupl	HF L-polym	HF L-sil	HF L-acid
EVA28 MFI=3 (ELVAX 265)	24	24	24	12	24	24
POE MFI=1,1 (Queo 8201)				12		
mLLDPE MFI=3-5 (EXCEED 3518)	10	10	12	10	8	8
LLDPE-g-MAH (FUSABOND E226)	4	4	1	4	4	4
Silane VTMOEO + DCP			1			
Hydrophobic agent (AR1055)					2	
fine pp ATH (MartinalOL104LEO)	60	58	58	58	58	58
Hydrotalcite (HT)						2
Silicon MB (AL1142A plus)	1,5	1,5	1,5		1,5	1,5
Stabilizer (AE1527E)	0,5	0,5	0,5	0,5	0,5	0,5
Dellite CW9		2	2	2	2	2

In the HF L-coupling formula, the coupling type was modified, partially replacing the maleic coupling (LLDPE-g-MAH) with a silane coupling (a VTMOEO+DCP blend). In the HF L-polymer formula, half of the EVA was replaced with POE to reduce the acidity of the polymer system. In the HF L-silane formula, a hydrophobicizing agent (AR1055) was added to make the system more basic. In the HF L-acidity formula, 2% hydrotalcite (acid buffer) has been added again to reduce the acidity of the system.

Table 30: Mechanical results of different nanocomposites for ageing studies

Mechanical Results	HF L-0	HF L-CW9	HF L-coupl	HF L-polym	HF L-sil	HF L-acid
Density at 23°C (g/cm^3)	1,480	1,454	1,477	1,452	1,457	1,454
MFI-21,6kg @160°C (g/10 min)	6,3	8,6	0,5	3,2	8,4	7,1
Tensile strength (MPa)	16,0	14,8	14,1	14,8	12,8	13,0
Elongation at break (%)	212	195	193	231	210	218
Tensile strength at 100% Elongation (MPa)	14,3	12,8	13,8	13,6	11,9	12,3
Tensile strength at 200% Elongation (MPa)	16,3	14,9	-	15,4	13,0	13,5

Table 31: Mechanical results after thermal ageing in air of different nanocomposites for ageing studies

Thermal ageing [168h @135°C in air]	HF L-0	HF L-CW9	HF L-coupl	HF L-polym	HF L-sil	HF L-acid
Tensile strength (MPa)	13,8	14,6	16,3	13,1	10,4	14,8
Elongation at break (%)	157	117	164	208	124	131
Tensile strength at 100% Elongation (MPa)	13,5	14,4	15,0	12,8	10,4	14,7
TS difference (%)	-14	-1	16	-11	-19	14
E@b difference (%)	-26	-40	-15	-10	-41	-40

After one week (168 h) at 135 °C the reference shows a 26% decrease in elongation, consistent with a recipe of this type. The HF L-coupling and HF L-polymer formulas showed small differences in post-aging elongation at break, indicating that the addition of Dellite® CW9 to a formula with silane coupling and **EVA-POE does not worsen the post-aging mechanical properties.** The HF L-silane formula again shows a 40% loss in post-aging elongation at break, indicating that the addition of a hydrophobicizing agent does not improve the thermal aging behavior of the material. The HF L-acidity formula with 2% hydrotalcite also showed a 40% loss in post-aging elongation at break, indicating that the addition of the acid buffer does not improve the thermal aging behavior of the material.

It is also important to evidence that the double coupling led to a very stiff blend with a MFI of 0,5 g/10min.

Table 32: Flame behaviour of tested compounds

Vertical burning test	HF L-0	HF L-CW9	HF L-coupl	HF L-polym	HF L-sil	HF L-acid
Time 1. Top of the flame to high limit (s)	67	103	200	168	152	165
Time 2. Flame base to graduation line (s)	171	244	385	307	531	345
Self extinguish (YES/NO)	NO	NO	NO	NO	NO	NO
Presence of burning drops (YES/NO)	YES	NO	NO	NO	NO	NO
Starting time of burning drops (s)	32	–	–	–	–	–
Flame height (cm)	>20	18	12	14-15	13-14	14-15
Flame width at graduation line (cm)	8-9	7	5-6	9	7	7

As it is clear from table 32, all the nano-compounds showed good flame behavior.
Following the results above, that established that the use of a mixed maleic-silane coupling improves the thermal aging stability of the formulation but decreasing MFI, in the below HF LC 2 formula, the maleic coupling (LLDPE-g-MAH) was still partially replaced with the silane coupling (VTMOEO+DCP blend), but at a lower dosage, compared to HF L-coupling.

Table 33: EVA/ATH based formulations for ageing studies

Eva based HFFR compound	HF L-0	HF L-C 2	HF L-F 1	HF L-F 2	HF L-stab 1
EVA28 MFI=3 (ELVAX 265)	24	24	24	24	24
mLLDPE MFI=3-5 (EXCEED 3518)	10	13	10	10	10
LLDPE-g-MAH (FUSABOND E226)	4	1	4	4	4
Silane VTMOEO + DCP		0,4			
fine pp ATH (MartinalOL104LEO)	60	58	56	56	58
$CaCO_3$ (Winnofils S)			2		
Silicon MB (AL1142A plus)	1,5	1,5	1,5	1,5	1,5
Silicon gum on silica (AQ1142 plus)				2	
Stabilizer (AE1527E)	0,5	0,5	0,5	0,5	0,5
Dellite CW9		2	2	2	
Dellite CW9 + 10% Stabilizers 1010/168 1:1					2

In the HF L-F 1 and HF L-F 2 formulas, the filler has been modified, in the first case with ultrafine $CaCO_3$ and in the second case with AQ1142 plus, a silicone rubber but supported on silica (powder). In the HF L-stab 1 recipe, a blend of organic stabilizers has been added to the Dellite.

Table 34: Mechanical results of different nanocomposites for ageing studies

Mechanical Results	HF L-0	HF L-C 2	HF L-F 1	HF L-F 2	HF L-stab 1
Density at 23°C (g/cm^3)	1,486	1,469	1,475	1,466	1,471
MFI-21,6kg @160°C (g/10 min)	5,8	6,8	6,0	3,4	7,8
Tensile strength (MPa)	15,8	10,2	12,4	15,8	12,5
Elongation at break (%)	224	209	230	233	222
Tensile strength at 100% Elongation (MPa)	14,4	11,0	12,9	13,1	12,0
Tensile strength at 200% Elongation (MPa)	16,1	10,1	13,1	14,1	12,8

As shown in table 34, all the compounds present good mechanical properties, even HF L-C2 (in yellow on table) has an acceptable tensile strength. Also, after one week at 100 °C, no significant drop in mechanical properties is visible.

Table 35: Mechanical results after thermal ageing in air of different nanocomposites for ageing studies

Thermal ageing [168h @100°C in air]	HF L-0	HF L-C 2	HF L-F 1	HF L-F 2	HF L-stab 1
Tensile strength (MPa)	15,4	9,8	11,6	13,6	12,4
Elongation at break (%)	199	183	216	195	219
Tensile strength at 100% Elongation (MPa)	14,6	11,4	12,2	13,3	12,2
TS difference (%)	-3	-2	-6	-14	-1
E@b difference (%)	-11	-8	-6	-16	-1

Table 36: Mechanical results after thermal ageing in air of different nanocomposites for ageing studies

Thermal ageing [168h @135°C in air]	HF L-O	HF L-C 2	HF L-F 1	HF L-F 2	HF L-stab 1
Tensile strength (MPa)	15,0	11,8	12,8	13,4	13,2
Elongation at break (%)	165	135	175	167	128
Tensile strength at 100% Elongation (MPa)	14,8	12,4	13,1	13,4	13,2
TS difference (%)	-5	18	3	-15	6
E@b difference (%)	-26	-35	-24	-28	-42

Moving to a heavier ageing, 135 °C for one week, the samples started to show some differences.
Mixing Dellite® CW9 with stabilizers didn't have any positive effect. The decreased silane coupling agent gave a good behavior after ageing without compromising the MFI. An optimization of the formula, with **0,5% of VTMOEO/DCP and 1% of FUSABOND E226 should give the expected MFI and resistance to ageing.**
In any case, **decreasing the ATH in favor of calcium carbonate or supported silicon gum gave the best results in terms of mechanical properties, MFI and ageing resistance.**

Conclusion

In conclusion, ageing both in water and air is an issue for all the nano-clays, ours and competitors. In a study not presented here, all our organically modified Dellite® grades and Argimel® B4C were tested and the results were comparable to the ones in previous chapter.

T**o overcome the water ageing and anti-hydrolysis agent could be employed** with ATH ora synthetic MDH. Natural MDH has a very high degree of hygroscopy that such stabilizers lose effectiveness. **Dellite® 72T does not require it because of its very low water uptake** compared to any other clay, ours and competitors, so it can be easily employed in any recipe, ATH or MDH based. This is very crucial for cable production as a high water absorption could change the electrical resistivity of a wire.
As a standard for air ageing in oven, Dellite® CW9 was considered due to its poor retention of mechanical properties at 135 °C for 1 week. After latest studies there are several option to overcome thermal ageing:

- Reducing acidity of the compound by **switching half of the EVA with POE**. In particular, 24% of EVA to 12% EVA and 12% of POE were evaluated.
- Partially reducing the **maleic coupling by substituting it with silane coupling.** In the study 4% of LLDPE-g-MAH changed to 1% of LLDPE-g-MAH and 0,5% of silane coupling agent showed the best performance.
- **Cutting 2% of ATH with calcium carbonate or supported silicon gum.** As presented 58% of ATH to 56% of ATH and 2% of CaCO3 or silane gum.

7. NON MODIFIED BENTONITE BASED PRODUCTS WITH FOCUS ON NEW DELLITE® EFB IN EVA BASED HFFR COMPOUD

For this study all compounds were made in a hot roll mixer. The applied temperature was 150-160 °C, for approximately 10 minutes. Previous studies showed that 2% of inorganic Dellite® does not work properly so this amount is increased to 10%.

Table 37: EVA based compound for filler comparison

Eva based HFFR compound	HFC LO	HFC LOO	HFC L1
EVA28 MFI=3	24	24	24
mLLDPE MFI=3-5 (Exceed 3518)	6	6	6
LLDPE-g-MAH (Fusabond E226)	4	4	4
fine pp ATH (MartinalOL104LEO)	54	54	54
Silicon MB (AL1142A plus)	1,5	1,5	1,5
Stabilizer (AE1527E)	0,5	0,5	0,5
$CaCO_3$	10		
Talco		10	
Dellite EFB			10

What can be observed in the table below is Dellite® EFB is the one that most influenced the rheology of the CaCO3 compound, halving the MFI.

Table 38: Mechanical results of HFFR based compounds

Mechanical Results	HFC LO	HFC LOO	HFC L1
Density at 23°C (g/cm3)	1,545	1,549	1,531
MFI-21,6kg @ 160°C (g/10min)	4,5	3,6	2,5
Tensile strenght (MPa)	10,5	10,7	12,2
Elongation at break (%)	165	113	155
Tensile strenght at 100% Elongation (MPa)	10,2	10,8	11,9

In table 38 it is also possible to observe that the bentonite gives a higher reinforcement than the reference recipe, compromising only a small amount of elongation, within a 10% decrease. **The formulation with Dellite EFB performs better than the compound with talc.**

Table 39: Mechanical properties after thermal ageing in air

Air Aging 7gg 100°c	HFC LO	HFC LOO	HFC L1
Tensile strenght (MPa)	11	12,2	12,3
Tensile strenght loss (%)	5	14	1
Elongation at break (%)	118	101	104
Elongation at break loss (%)	-28	-11	-33
Tensile strenght at 100% Elongation (MPa)	10,8	12	12,1

Table 39 and 40 show that bentonite performed overall similarly or better than the Talc and $CaCO_3$ references, both in terms of load and elongation, in all aging processes.

Table 40: Mechanical properties after thermal ageing in water

Water Aging 7gg 100°c	HFC L0	HFC L00	HFC L1
Tensile strenght (MPa)	9,3	9,1	8,7
Tensile strenght loss (%)	-11	-15	-29
Elongation at break (%)	119	102	135
Elongation at break loss (%)	-28	-10	-13
Tensile strenght at 100% Elongation (MPa)	9,3	9,3	8,7
Weight gain in Water [%]	0,223	0,279	1,502

Must be said that formula with Dellite® EFB absorbs 1.5% water after aging, indicating a high hygroscopicity.

Table 41: Flame behaviour of tested samples

Vertical burning Test [1mm]	HFC L0	HFC L00	HFC L1
Time 1. Top of the flame to high limit	152	131	156
Time 2. Flame base to graduation line	240	227	248
Self extinguish	NO	NO	NO
Presence of burning drops	YES	YES	NO
Starting time of burning drops	38	63	–
Flame height	15-16	18-19	17-18
Flame width at graduation line	5	10	9

The flame results show that Dellite® EFB improves the compound’s flame behaviour. It does not drip during vertical burning.

Conclusion

The introduction of Dellite® EFB in the EVA-ATH compound in place of $CaCO_3$ improves the material’s flame properties, **preventing dripping during the burn test.**
Mechanical properties remain good even with the introduction of bentonite in place of $CaCO_3$ and are **even better than Talc.**
Water aging showed good results compared to reference, but the hygroscopicity of Dellite® EFB may lead to a problem in electrical tests, since **it absorbs a lot of water during ageing.**

8. NON MODIFIED BENTONITE BASED PRODUCTS WITH FOCUS ON NEW DELLITE® EFB IN PVC BASED COMPOUND AS SUBSTITUTION TO MDH

The following report analyzes the behavior of Dellite® EFB as a substitute for magnesium dihydroxide (MDH) in PVC-Flame Retardant cable formulations. As a starting point, different bentonite based nanocomposites have been compared to the reference formulation with MDH (PVC L-0). Various properties have been evaluated, with particular attention to colour.

All compounds have been manufactured in a co-rotating twin-screw extruder and subsequently processed in a hot-roll mixer. Specimens have been obtained by molding and hot-press shaping. Processing temperatures were always between 160 and 170 °C.
For color evaluation, flat-head single-screw extrusions were also performed.

Table 42: Starting formulations for PVC based compound study

PVC based Formulation	PVC L-0	PVC L-9	PVC L-12	PVC L-13	PVC L-14
PVC resin (K70 inovyn)	41	41	41	41	41
DIDP	21	21	21	21	21
$CaCO_3$ (Omyacarb 2T-AV)	21	21	21	21	21
MDH	12				
Dellite EFB		12			
Dellite® EFB act 10% Na-stear			12		
Dellite® EFB mix 10% Na-stear				12	
Dellite® EFB mix 10% Stearic ac					12
ATO	2	2	2	2	2
Zn Borate	1	1	1	1	1
Ca/Zn Stabilizer CZH37V7	2	2	2	2	2

Different bentonites and different coating/mixing were taken into account as it is shown in table 43.

Table 43: Thermal and physical properties of PVC based compounds

Properties	PVC L-0	PVC L-9	PVC L-12	PVC L-13	PVC L-14
Thermal stability (CR 200 °C)	90	78	62	70	88
LOI	31	32	29	29	31
UL-94 Classification	-	V0	-	-	-
Density at 23°C (g/cm³)	1,51	1,52	1,51	1,50	1,51
MFI -21,6kg @170°C (g/10 min)	9,1	2,2	-	-	-
Hardness ShA	88	93	90	89	92

As it is clear from table 43 and 44, adding 12% Dellite® EFB, in comparison to MDH, does change the MFI (Melt Flow Index) without significantly compromising other mechanical properties. It is possible to observe a good improvement in terms of tensile strength.

Table 44: Mechanical properties of PVC based compounds

Mechanical Results	PVC L-0	PVC L-9	PVC L-12	PVC L-13	PVC L-14
Tensile strength (MPa)	11,9	14,6	13,6	11,3	11,1
Elongation at break (%)	181	150	168	192	120
Tensile strength at 100%	9,0	13,8	11,4	9,0	10,5

The LOI (Limiting Oxygen Index) results in table 44 show that the **Dellite® EFB performs as well as MDH during the test, with a slight increase from 31 to 32.**

Bentonite reduces the thermal stability of compounds, but this contrasting behavior is damped by this Dellite® composition. The loss is limited and the material shows an acceptable Cong Red test.

Regarding UL-94 test, all tested samples consistently scored V0, with increased char formation for **Dellite® EFB** based compound.

Regarding mechanical properties, they were also evaluated for an extruded sample, without processing in a hot roll mixer.

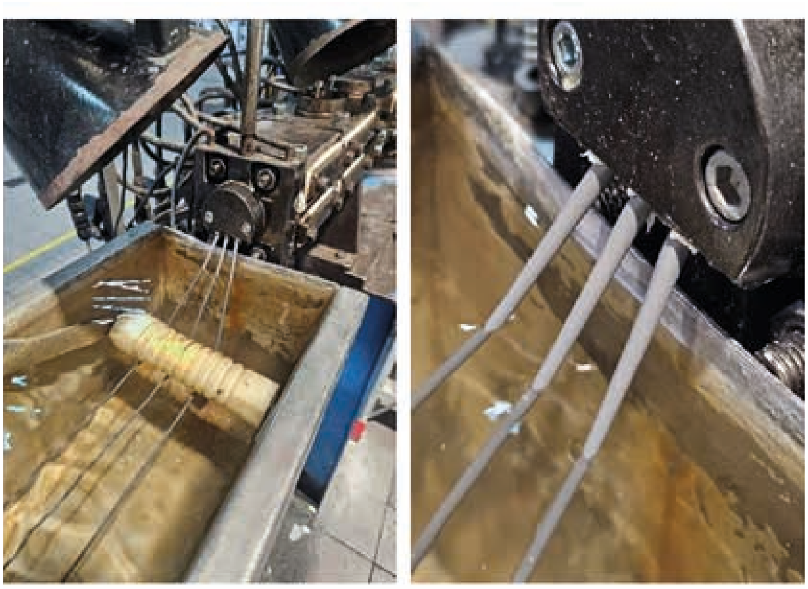

Figure 13: Extrusion before water-cutting of Dellite EFB based compound (PVC L-9).

Despite the employed bentonite is possible to observe that a complete flattening of mechanical occurred. In particular all the samples behave similarly to MDH in terms of tensile strength and elongation at break.

Table 45: Process parameter and mechanical results after extrusion.

Process of extrusion	PVC L-0	PVC L-9	PVC L-10	PVC L-16
Temperature	170-170	170-170	170-170	170-170
T°C max	176	177	176	175
Bar	25	30	22	21
Torque	5	5	4	4
Amp	7.4	7.9	6.9	6.8
Rpm	200	200	200	200
Feeder	15	15 (7)	7	7
Tank	Normal	Normal	Normal	Normal
Pelletizer	STD	STD	STD	STD
Degassing	2	2	2	2
Bridge break	Sì	Sì	Sì	Sì
Twin screws	STD	STD	STD	STD
Mechanical Results				
Tensile strenght (MPa)	14	13,8	13,4	15,2
Elongation at break (%)	158	153	148	145
TS at 100% of Elongation	11,7	12,3	12	14,1

It is also clear from Figure 13 and 14 that the color tends to grey while employing Dellite® EFB in PVC resin.

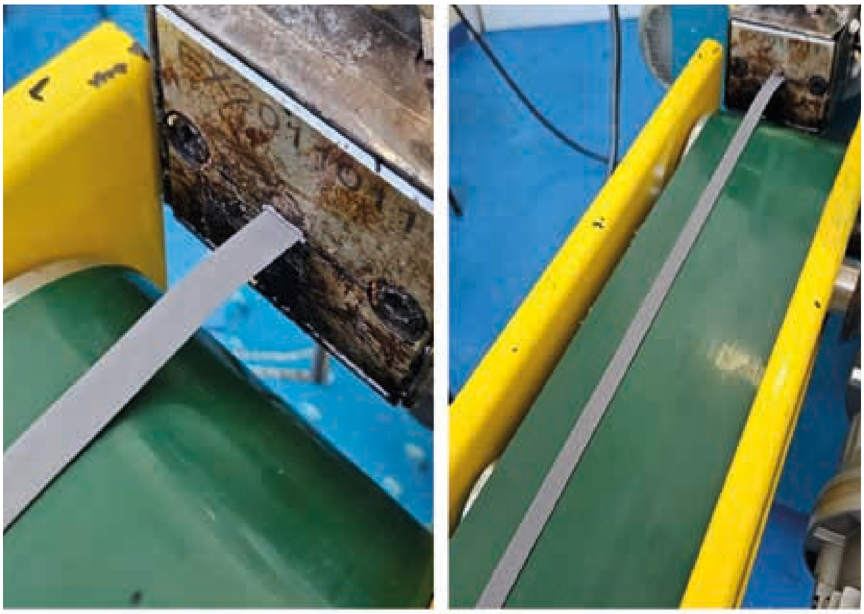

Figure 14: Flat-headed mono-screw extrusion of PVC L-9 compound.

For Dellite® EFB containing compounds, color is schematized in the next table.

Table 46: Colour pattern schematization for PVC L-9 and its variations.

PVC based Formulation	PVC L-0 (phr)	PVC L-9 (phr)	PVC L-17 (phr)	PVC L-26 (phr)
PVC resin (K70 inovyn)	100	100	100	100
DIDP	50	50	50	50
$CaCO_3$ (Omyacarb 2T-AV)	50	50	50	50
MDH	30			
Dellite EFB		30	30	30
ATO	5	5	5	5
Zn Borate	2	2	2	2
Ca/Zn Stabilizer CZH37V7	4	4	4	4
TiO_2			3	
MB Blu HX2247				8
Final colour	White	Grey (dark)	Grey (light)	Blue

In order to deal with colour, a new formulation was developed.
Instead of PVC resin, an extruded granule was employed, containing resin and plasticizer.
Avoiding the use of dry blend, switching to an already worked material leaded to results in table below.

Table 47: Recipe variations to avoid greying of the material.

PVC based Formulation	PVC L-21 (phr)	PVC L-22 (phr)	PVC L-23 (phr)	PVC L-24 (phr)
PVC Granule	150	150	150	150
$CaCO_3$ (Omyacarb 2T-AV)	50	50	50	50
Dellite EFB	30	30	30	30
ATO	5	5	5	5
Zn Borate	2	2	2	2
Ca/Zn Stabilizer CZH37V7	4	4	4	4
MB Blu HX2247		8		
MB Marrone LX1661			8	
MB Verde IX2221				8
Final colour	White	Blue	Brown	Green

As it is possible to understand by Table 47 no greying was observed, and the final color of compounds was clear, without variation due to a grey base.

Figure 15: PVC L-9 and PVC L-21 comparison. The new formulation presents no grey colour

Conclusion

In conclusion, bentonites can replace MDH in PVC-FR formulations, offering competitive pricing and comparable mechanical and hardness properties. Some bentonites can achieve also good thermal stability. All non-modified bentonite based compounds reached a **consistent V0 classification.**

In flame tests, **Dellite® EFB performed best in terms of LOI**, but it also had an effect on the color of the compound. It was chosen because, all the differences in mechanical behavior are flattened after a twin-screw extrusion. PVC compounds are very influenced by the way they are worked.

This color generation can be covered with proper masterbatches, or it can be **successfully avoided by switching from dry mix compounding to an already produced PVC/plasticizer masterbatch.**

9. DELLITE® SYNERGIES WITH INTUMESCENT PP BASED SYSTEMS

The following test analyses the performance of Dellite® as a partial replacement and synergistic additive with intumescent systems currently available on the market. The chosen system was Exolit AP766, one of the best intumescent system for PP.
Dellite® grades, added at 2% by weight, allows for a reduction in the total intumescent system dosage while maintaining the same UL-94 rating.

Table 48: PP based compound for intumescent synergy study

PP based compounds	PPL-0	PPL-2	PPL-3	PPL-4	PPL-5	PPL-7
PP homo grado 8	75	77	77	77	77	77
Exolit AP766	25	23	21	21	21	21
Dellite 72T			2			
Dellite CW9				2		
Dellite 67G					2	
Dellite 43B						2

Below flame behaviour is schematized.

Table 49: Flame behaviour of tested compounds

Fire results	PPL-0	PPL-2	PPL-3	PPL-4	PPL-5	PPL-7
UL-94 classification (1,6mm)	V0	V2	V2	V2	V2	V0
Total burning time (s)	10	20	65	20	45	42
Higher post combustion time (s)	1	4	13	5	7	8
Dripping 1st application (secs)	NO	NO	NO	NO	NO	NO
Dripping 2nd application (secs)	NO	YES	YES	YES	YES	NO
Dripping 3rd application (secs)	YES unf *	YES	YES/NC	YES	YES	YES

UL-94 test results in table 49 clearly show that Dellite® 43B and is the most suitable for this application not changing the V0 classification of the starting compound.

Conclusion

In PP based systems **4% of Exolit AP766 can be successfully replace by Dellite® 43B** without losing V0 classification in UL-94 standard test.

10. DELLITE® SYNERGIES WITH INTUMESCENT OIL EXTENDED SEBS SYSTEMS

The following test analyses the performance of Dellite® as a partial replacement and synergistic additive with intumescent systems currently available on the market. The chosen system was Exolit AP766, the best intumescent system for oil extended SEBS.
Dellite® grades, added at 2% by weight, allows for a reduction in the total intumescent system dosage while maintaining the same UL-94 rating.

Table 50: SEBS based compounds for intumescent synergy study

Oil extended SEBS based compounds	ISL-1	ISL-2	ISL-3	ISL-11	ISL-14
SEBS Kraton	30	31	31	32	32
Oil (Ramoil)	30	31	31	32	32
Adsyl 5C 37F	8	8	8	8	8
Exolit AP766	32	30	28	26	26
Dellite 72T			2	2	
Dellite 43B					2

As it is possible to understand from table 51 only Dellite® 72T gave the same class of the starting compound lowering the amount of Exolit AP766 by 4%. This is a very fore generating system and, lowering too much the intumescent portion leaded to a non classification in UL-94 test. ISL-11, with 6% of intumescent cut ganarated dripping burned drops at first application.

Table 51: Flame behaviour of tested compounds

Fire results	ISL-1	ISL-2	ISL-3	ISL-11	ISL-14
UL-94 classification (1,6mm)	V0	V2	V0	NC	NC
Total burning time (s)	20	61	25	–	–
Higher post combustion time (s)	6	17	5	–	–
Dripping 1st application (secs)	NO	NO	NO	YES	NO
Dripping 2nd application (secs)	NO	YES *	NO	–	NC
Dripping 3rd application (secs)	NO	YES	NO	–	–

Conclusion

In SEBS based systems **4% of Exolit AP766 can be successfully replace by Dellite® 72T.**

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