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Compounding WORLD

DEVELOPMENTS IN FIRE RETARDANTS

COMPOUNDING IN THE LABORATORY

KEEPING COUNTERFEITERS IN CHECK

IMAGE: SHUTTERSTOCK

Regulation drives FR development

The safety benefit of flame retardants is clear but tighter health and environmental regulation is pushing development in the direction of more sustainable solutions. Peter Mapleston reports

Regulation and consumer preference are proving the key drivers behind development of new flame-retardant systems for use in thermoplastics. Suppliers of an increasingly diverse variety of halogen-free flame retardants (HFFRs), which are increasingly seen as more sustainable than halogenated types, are making considerable progress in addressing performance issues relating to some of their products. Meanwhile, producers of traditional and long-established brominated systems (BFRs) are presenting arguments to challenge what they see as unfair new laws restricting their use.

On the halogen-free side, industry association **PINFA** (Phosphorus, Inorganic & Nitrogen Flame Retardants Association) says it sees interest in HFFRs continuing to grow. "Strong opportunities, or indeed obligations, for PIN FRs will result in coming years from the EU's Green Deal," it says. "The Green Deal is already leading to new policy initiatives such as the new Chemicals Strategy with its 'Zero Pollution Ambition,' and the Sustainable Products Initiative. Further policy developments are expected soon, including a revision of RoHS (Restriction of Hazardous Substances), which already bans several brominated FRs in E&E applications."

HFFR take-up is also expected to benefit from EU moves towards circularisation and ecological design. "The new Circular Economy Action Plan specifically targets recycling in electronics and IT, batteries and vehicles, plastics and textiles so that plastics additives must aim to be safe for end-of-life recycling," according to PINFA. "The EcoDesign Directive [will exclude] halogenated FRs in enclosures and stands of screens and displays. Proposals are being considered to widen the scope of the Directive, which currently covers only certain electrical products."

The new EU policy context will pose challenges to the chemicals industry and to compounders and plastics users. But PINFA believes it will also bring opportunity for innovation, pointing out that nearly all R&D in the area of flame retardancy is today targeting PIN FRs. "The trend towards environmentally preferable PIN FRs will be accelerated," it says. "At the same time, existing PIN FRs, known to be safe and effective, will continue to be improved.

Main image: Tightening regulation, particularly in Europe, is challenging flame retardant producers to meet increasingly demanding environmental and performance goals

New formulations, co-polymers or oligomer forms, combination packages of PIN FRs or use of specific PIN synergists, will ensure that PIN FRs respond to demanding user specifications."

It should be said that PINFA's perspective contrasts with that of **BSEF** (the International Bromine Council) whose members include leading suppliers of brominated flame retardants. BSEF has lodged formal proceedings with the European Court of Justice in Luxembourg against the EC with respect to the new regulation on electronic displays. Keven Bradley, Secretary General of the BSEF, says it disagrees with the regulation's "unprecedented and unwarranted restriction." See the article on page 35 for more information on this.

Automotive drivers

PIN FR additives producer **Adeka** sees automotive safety concerns driving interest forward. "Increasing safety awareness is driving expanded use of flame retarded materials," the company says. "In automobiles, flame retarded materials have until recently only been used for interiors. For the next generation of automobiles, however, flame retarded materials are expected to be used for the entire propulsion system, including the various parts of battery construction in an EV. Less obvious but just as crucial, flame retarded materials are expected to be used for high voltage equipment including charging stations and electronic instruments for control and operation."

Flame retarded PC and PC alloys are being considered for charging station housings for EVs, Adeka says. "Currently retardancy performance requires a UL94 V-0 classification at 1.6mm. The trend however is toward V-0 at 0.8 and even 0.4mm."

Bisphenol-A bis(diphenyl phosphate), otherwise known as BDP and which Adeka supplies as ADK Stab FP-600, and resorcinol bis(diphenyl phosphate, or RDP, are both commonly used as flame retardants for PC and PC alloys. But Adeka says they have some limitations. "In order to meet the flame retardancy requirements at lower thickness, higher

Left: **Car makers are facing flame retardant selection challenges, including compliance with new EV standards and regulations, calls for improved sustainability, and the need to avoid corrosion of electronic and electrical systems**

IMAGE: PINFA/SHUTTERSTOCK

IMAGE: SHUTTERSTOCK

Above: FRX Polymers' Nofia can provide flame retardance in PC alloys without compromising car makers' preferred piano black finish

loadings of the flame retardant will be required. The decrease of heat deflection temperature (HDT) accompanying this makes the flame retarded material unsuitable for certain applications."

To solve this problem, Adeka has developed a new liquid phosphate ester flame retardant – ADK Stab FP-900L – that it says combines higher flame retardancy and an improvement of HDT compared to market reference phosphate ester FR. The improved HDT comes from the combination of the reduced plasticising effect of the additive due to its more rigid oligomeric structure as well as the lower dosage levels required, the company says.

In tests with a PC reinforced with 10% glass, ADK Stab FP-900L dosed at 20% provided a V-0 classification at 0.8mm, whereas BDP could only achieve V-2. In a 30% reinforced sample, the same result was achieved at an FP-900L addition level of 15%. In both formulations, HDT was around 5°C higher for the compound based on the new flame retardant.

Flame retardants for PC, as well as other thermoplastic polyesters, are available from **FRX Polymers**. At last year's K2019 trade show, the company introduced developments with its Nofia polymeric and reactive phosphonate products in applications for recycled PET (rPET) and in PET/PC and PBT/PC blends for medical applications. Updating on progress this October, Sales & Marketing VP Ina Jiang said PC/PBT blends have been receiving OEM approvals in medical and also automobile interior applications. She says that some customers have also carried out trials with PC/ABS and PC/ASA blends, which have "an excellent overall balance of properties and exceptional processing." In these blends, it has been possible to obtain V-0 ratings down to 0.75mm. "This is outstanding for a

No	Composition [%]			FR Testing (1.6mm)	Transparency [1mm]	Izod Impact Notched [-30°C]
	PC	Anti-drip	SFR320	UL94	[%]	[KJ/m²]
1	100	0	0	V2	89	12
2	99.85	0.15	0	V2	88	17
2	99.85	0.15	1	V0	88	14

Table 1: Momentive's SFR320 flame retardant can deliver a UL94 V-0 rating in polycarbonate at 1% concentration while maintaining transparency and low temperature impact properties. Momentive says the phenyl substituted low viscosity silicone fluid (previously marketed as Y-1932) is compatible with many polymers and can provide improved processability, flame propagation inhibition and combustion rate
Source: Momentive

PC alloy," says Jiang. "Most PC alloys obtain V-0 at no thinner than 1.5-1.6mm."

Jiang sees good potential for PC/PBT compounds containing Nofia in automotive interiors, since it is possible to obtain parts with the car industry's preferred high-gloss piano black finishes. She also notes the unwanted plasticising effect of BDP and RDP in PC alloys. "In a non-FR PC/ABS system, the HDT is around 120-130°C and BDP/RDP can reduce that to 85°C. The Nofia solution has no effect on the HDT. In PC/PBT, the HDT of a Nofia-based solution is 30°C higher than BDP/RDP solution."

Nofia has already shown advantages in PET fibre and rPET, but Jiang says PET films and foams are now also proving to be key applications. In BOPET film for example, she says Nofia is the only flame-retardant system that can yield the highest UL94 VTM (Vertical Thin Material) rating of VTM-0 in a fully transparent film down to 15 microns. Other halogen-free FR additives can cause haze. The Nofia flame retardant can be added directly into the film extruder or used in a masterbatch.

Developing potential

Other key applications for VTM-0 BOPET films include 5G components, EV batteries, numerous electronic devices, high-speed data transmission flat cables, and solar panels. The company says Nofia polymeric FRs can also function as masterbatch carriers. As a lot of BOPET film requiring FR properties also needs to be black, the additive can be used as a carrier for carbon black, which Jiang says can result in a cost saving.

Claiming to be the world's largest supplier of sulphonate FR products for polycarbonates, **Arichem** says the technology is "uniquely suited to PC." It says its products are specific to the chemistry of how PC reacts to fire and are most often used at addition rates of less than 1%. The company says that its Arichem KSS-FR grade is already REACH registered and other Arichem FR products are in the process of registration.

"Arichem's FR products answer the call for halogen-free and are neither phosphorus nor metal hydrate based," says the company. "With the recent inclusion of potassium perfluorobutane sulphonate (and higher alkyl analogues) in the EU SVHC listing, reformulations with Arichem KSS-FR are taking place at an increasing pace across the FR PC compounding industry."

According to **Momentive**, customers in many industries using FR plastics are seeking alternatives to traditional halogenated flame retardants in response to a wave of new regulations in both Europe and the US that seek to address several related concerns covering environment, health, safety and recycling. "Given these trends, silicone-based solutions appear attractive from both a performance and low toxicity point of view. In terms of the former, the silicone-oxygen-bond offers certain distinct advantages over more traditional carbon-carbon-bond based approaches, most importantly when it comes to maintaining good mechanical properties and flame retardancy characteristics," the company says.

"The greater length of the Si-O bond enables greater permeability, the higher bond energy allows better stability, and the lower rotational barrier offers more flexibility. Combined, these differences lead to lower surface energies, better moisture, temperature and oxidation resistance, UV stability, biocompatibility and insulation properties," Momentive claims.

At **Techmer PM**, Product Development Manager Kaan Serpersu says the company is seeing increased demand in its key non-halogen and Oeko-Tex approved flame-retardant technology for the PET fibres market. "Traditional polymeric flame retardants for PET create numerous challenges in processing issues, mainly because of the low melting point of the flame retardant," he says. "Techmer PM's PTM112508 technology widens the process window allowing products to meet the most stringent specifications."

Serpersu says the technology, originally designed to meet the rigours of military apparel applications in accordance with ASTM D6413, can be designed for other specifications such as NFPA 701 and UL94. It is available in pellet form for easy implementation across various production processes.

Huber Engineered Materials says it has been advancing its FR business on several fronts. First off,

IMAGE: SHUTTERSTOCK

Above: The cable industry is a key market for halogen-free flame retardant solutions, with increasingly demanding standards being imposed

it is in the process of introducing a new Safire nitrogen-phosphorus fire retardant technology. Secondly, it has also provided new data for its Kemgard smoke suppressants for use in flexible and rigid PVC. And thirdly, the company's Martinal OL-104 LEO precipitated ATH – originally developed in Germany by Huber Fire Retardant Additives business Martinswerk – has now been available in North America from the company's plant at Bauxite, South Arkansas.

Huber says Safire 400 melamine poly(zinc phosphate) "imparts an enhanced balance of flame retardancy, smoke suppression and char formation. It has been designed to catalyse the formation of a strong glassy char layer in most thermoplastics. In addition, the presence of zinc further reduces smoke production and helps suppress arc track values when required."

In recent studies, the company says Safire 400 has been shown to be a highly efficient synergist with aluminium hydroxide in LSZH (low smoke zero halogen) cable formulations. Partial replacement of AlOH reduces the rate of polymer decomposition via char formation. The protective layer of char provides a heat-insulation effect, reduces oxygen access, and prevents dripping of molten polymer. "Furthermore, the potential to reduce overall filler content will result in better physical properties of the jacketing compound," Huber says.

Kemgard smoke suppressants comprise molybdates precipitated on a functional core material to maximise the active surface area. Depending on the material, the core can offer secondary benefits such as additional fire retardancy or improved processability. The molybdate chemistry is designed to catalyse crosslinking in the PVC matrix, leading to improved organic char formation.

Cable demands

Germany's **Nabaltec** says that polyolefin compounds highly filled with aluminium hydroxide (ATH), which it sells as Apyral, or magnesium hydroxide (MDH) are widely used for sheathings of electrical and communication cables. However, for low fire hazard and fire-resistant cables more stringent requirements are imposed and these standards can be difficult to meet and frequently render the additional incorporation of flame-retardant synergists necessary, the company says.

Certain synergistic flame retardants (such as organically modified nanoclays) can negatively impact compound processability and aging performance, according to Nabaltec. To overcome this problem, it says it has developed a new flame-retardant booster – Actilox PA-B2 – that it says allows HFFRs to fulfil demanding flame retardancy standards while providing enhanced compound processability.

"Furthermore, Actilox PA-B2 leads to a reduced

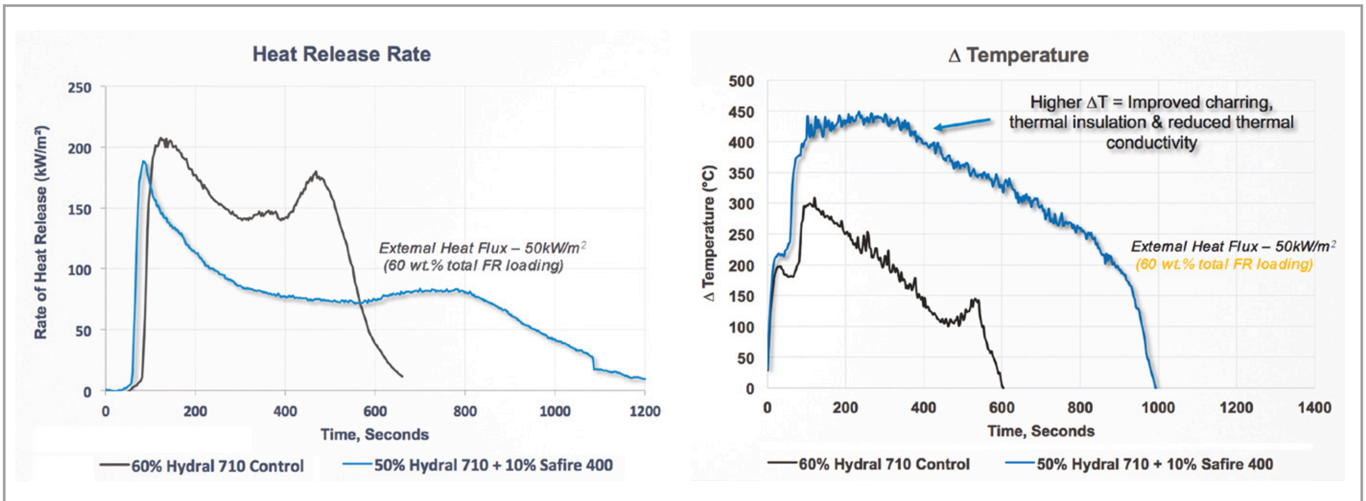


Figure 1 and 2: Graphs showing heat release and difference in temperature between sample upper surface and underside in cone calorimeter testing in a cable compounds containing Huber's Hydral 710 precipitated alumina trihydrate and Safire 400 nitrogen-phosphorus fire retardant

Source: Huber Engineered Materials

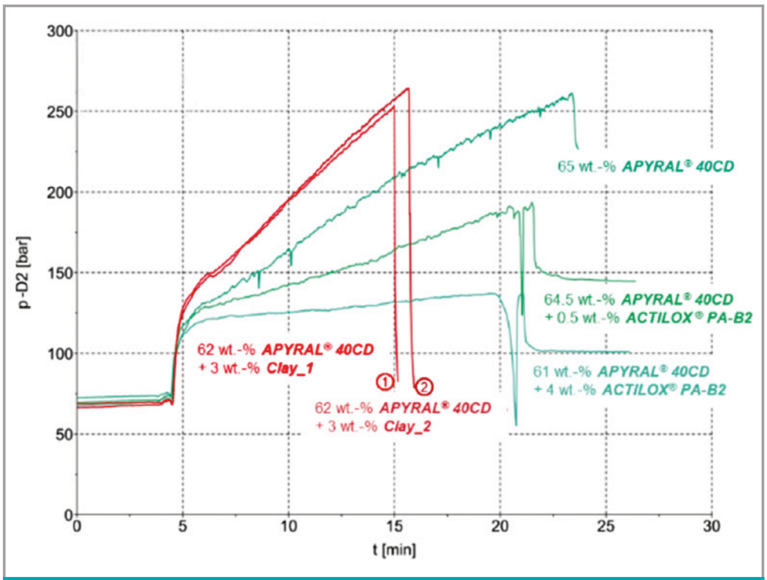


Figure 3: Pressure build-up over time of different flame retardant LLDPE/EVA formulations using the DIN EN 13900-5 filter pressure test. This determines the quality of the filler dispersion in the polymer matrix and the influence of mineral fillers on the pressure build-up during compounding and extrusion due to blocked screens. Compound obtained from the twin screw extruder was melted in a single-screw-extruder and conveyed through a 50µm filter. The pressure build-up in front of this filter was measured and evaluated.
Source: Nabaltec

pressure build-up during compounding and extrusion while maintaining a good mechanical and aging performance," it says. Nabaltec has summarised the features of Actilox PA-B2 in a **White Paper**, which is available on its website.

Russian Mining Chemical Company says it sees a growing number of aluminium composite panel (ACP) producers looking to move from B1/B2 (flame retardant) class panels to A2 (incombustible) class in accordance with EN13501-1. "During the last year we have received several times more requests for EcoPiren (based on Brucite, the mineral form of magnesium hydroxide) suitable for production of A2 core compounds in comparison with the year before. The main ACP products cluster is still rated as B1/B2, which is produced using the conventional extrusion technology," the company says.

"There is still a strong belief that an A2 core compound cannot be produced by extrusion, especially with acceptable density (which is about 1.75g/cm^3). That's why we dedicate a lot of our R&D resources to develop new EcoPiren grades and optimise the formulation for A2 class core compound to provide expected level of processability and density by extrusion," it claims. An intermediate result of this work is a new EcoPiren 400-1000 grade for continuous compression lamination. It has already been approved by several leading producers of A2 ACP.

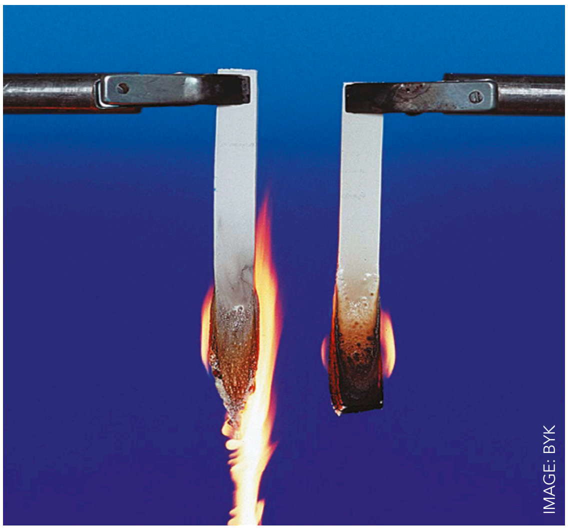
Quarzwerke's **HPF The Mineral Engineers** unit has developed what it says is an innovative flame-retardant material in cooperation with a well-known (but unnamed) automotive supplier and a large compounding company. HPF supported the project with various coated and uncoated Kaolin grades, with Kaolin TEC 110 EST showing the best overall performance in the compound. This is a platey phyllosilicate with a high aspect ratio. "With Kaolin TEC 110 we managed to develop a particular high aspect ratio quality with excellent reinforcing properties," says Péter Sebö, Head of Marketing & Market Development at HPF.

Sebö says the olefinic compound developed in the trials shows extremely good flame resistance and good long-term heat stability up to 150 °C. Mechanical properties, such as impact strength, are said to be excellent at temperatures down to -20°C. They were achieved using Kaolin TEC 110 in combination with various halogen-free additives. "In the event of a fire, no corrosive smoke gases are produced and the smoke gas development is hardly measurable," says Sebö.

The compound passed the glow wire test (GWT) at 750°C according to IEC 60695-2-10/11, with a 1.75mm penetration depth after 30 sec. At 800°C, penetration depth after 30 sec was 2.70mm. It achieves a UL94 V-0 rating and has very good resistance at 23°C and 60°C to various chemicals, including fuels and oils. Flammability tests were run with specimen thicknesses down to 0.8 mm. "We think that a V-0 below 0.8mm is also possible," says Sebö. The recipe and the precise loading of Kaolin is confidential, but is said to be more than 10%.

Targeting polyamides

Last year, **Byk** launched Byk-Max CT 4260, an organophilic sheet silicate for use in thermoplastics



Right: Fire performance of a PA compounds without Byk-Max CT 4260 (left) and with a 5% addition

such as polyamides. The company says the product is especially suitable for use with HFFR compounds "since incorporating the additive improves not only the flame retardant properties but also the dropping behaviour and crust formation."

Byk-Max CT 4260 also enables the filler content – aluminium, or magnesium hydroxide, for example – to be reduced. This leads to a measurable improvement in processing and physical properties and to a reduction in the overall weight, Byk says. It can also enhance barrier to oxygen, water vapour, and hydrocarbons while increasing melt viscosity, enabling in some cases an improvement in dimensional stability during profile extrusion.

At **Budenheim**, Heiko Rochholz, Head of Marketing, says with its new Budit 617, the company "offers a new class of HFFR system that addresses the need for a higher GWIT (>800 °C) and less corrosion while processing."

Budit 617, which is based on melamine polyphosphate in a new synergistic combination, is intended for use in glass fibre reinforced polyamide 66, where it can provide up to UL94 V-0 at 0.8 mm. It does not discolour the polymer and does not migrate to the part surface. Decomposition starts above 340°C. "Due to its high phosphate and nitrogen content it acts as a flame retardant mainly in the solid phase [char formation] but has additionally a synergist (DOPO derivative) acting in the gas phase," says Rochholz.

Budenheim is also now offering Budit 669S, a development of its existing Budit 669 grade that is suitable for use in polyolefins, especially cables, tubes, and housings of white goods. 669S is an ammonium polyphosphate (APP)-based halogen-free flame retardant containing a special coating. Compounds using it are said to offer better mechanical properties. In addition, there is no migration and V-0 can be achieved at 1.6mm.

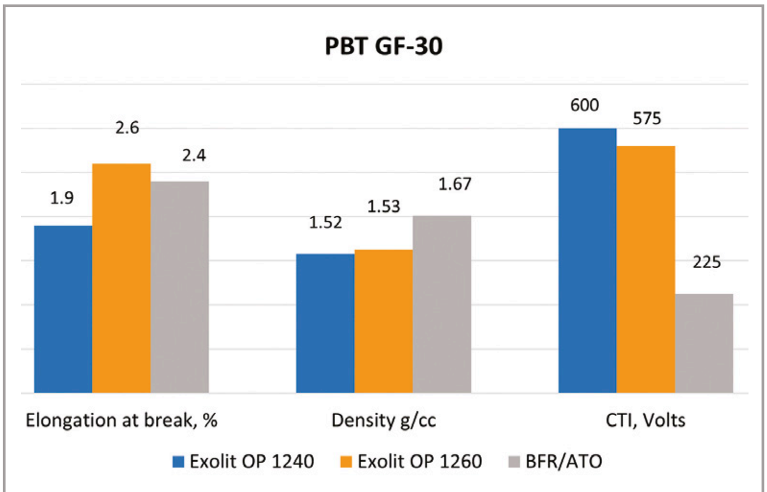


Figure 4: Clariant's Exolit OP 1260 flame retardant offers a good balance of properties in glass reinforced PBT compounds
Source: Clariant

Circular innovations

Addressing issues of the circular economy, **Clariant** says there is a growing need for flame retardants for polymeric compounds that are not only efficient and stable but also environmentally friendly. It says that for its Exolit halogen-free flame retardants for polyamides and polyesters, it has developed a rigorous method to screen products based on a catalogue of 36 criteria to determine their environmental impact. Based on this extensive evaluation process, products that meet the criteria can be awarded its Ecotain label. The company cites Exolit OP 1400 for polyamides as an example. It says it "combines sustainability with the ability to meet the stringent requirements of e-mobility like UL94 V-0 down to 0.4 mm and the best CTI of 600V."

For thermoplastic polyesters, Exolit OP 1260 has also been awarded the Ecotain label, in part due to good recycling performance demonstrated in a recent study of PBT 30% glass reinforced compounds with and without flame retardants. Specimens were moulded from fresh compound with the addition of 10%, 30% and 50% regrind. The Exolit-based compounds retained the flame retardancy standard UL 94 V-0 at all levels of re-grind addition. Even the afterburning times were not significantly affected, the company claims. The mechanical properties including elongation remained unchanged as well. "Under the conditions used in the test, Exolit OP 1260 can be used as a drop-in replacement to brominated flame retardants," says Clariant.

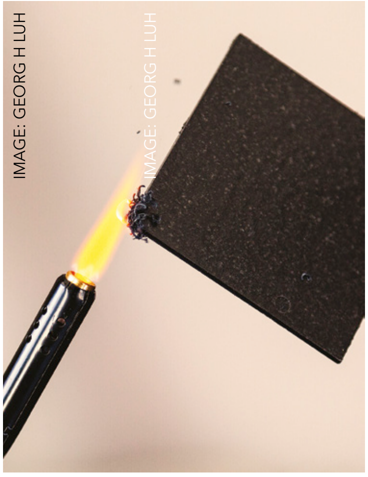
Progress is also being made in flame retardants based on graphite, with **George H Luh**'s new expandable graphite grade developed especially for polyamides. The company, which provides materials from various sources, says that, until now, the expansion initiation temperature of maximum 230 °C has limited the use of expandable graphite in polymers with relatively high melting points such as polyamide. "With our new generation expandable graphite GHL PX 95 HT 270, we have been able to develop a new grade that only develops its protective characteristic from 270°C," says Marketing Manager Angelina Schöffel. "It can be used for polyamides and applications with processing temperatures up to 260°C."

Quantities for sampling and first production trials are available now. The company says the next step is the development of expandable graphite grades with even higher starting temperature and various particle sizes.

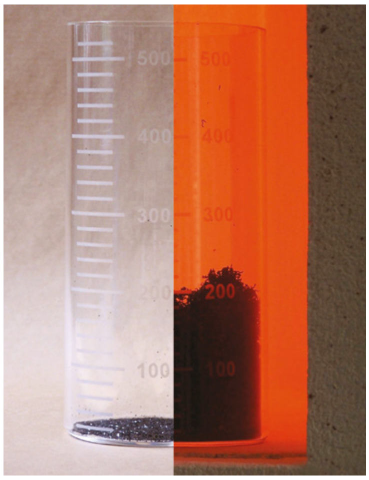
Expanding research

The fire protection effect of the new expandable graphite and its processing in various polymers is being investigated in a research project between Luh and the Institute of Polymer Technology at the University Erlangen-Nuremberg in Germany. Combinations with different synergists such as MDH and APP, as well as further optimisation potential of the expandable graphite itself, form part of the project. Preliminary studies have shown that the intumescent layer acts as a heat shield that reduces heat generation and slows fire progress. The Average Rate of Heat Release (AHRE) is significantly improved and combustion is near smoke-free.

NeoGraf Solutions has been manufacturing carbon and graphite products for more than 135 years. It specialises in the development and manufacturing of natural and synthetic graphite sheets and powders for numerous applications. It recently added GrafGuard 280-50N to its line-up of expandable graphite flake, non-halogenated flame-retardant additives. The new grade is being targeted at compounds for injection moulded and thermoformed applications.



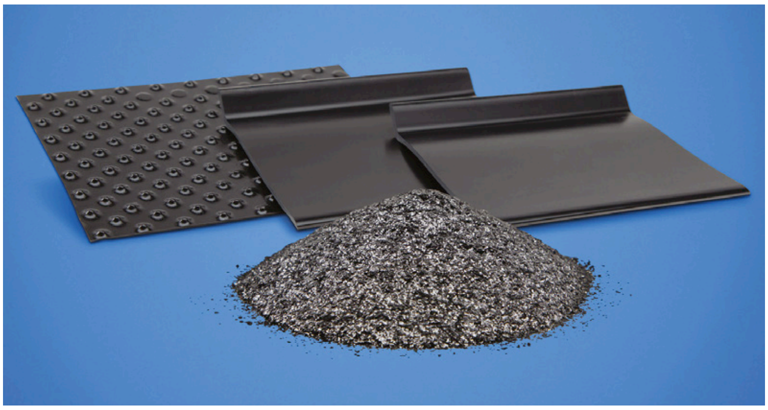
Above: A flame being put to a PA compound filled with expandable graphite



Above: Expandable graphite before (left) and after expansion (right)

With a particle size of 300 microns, GrafGuard 280-50N has an on-set (or expansion initiation) temperature of 280°C, which NeoGraf says is the highest on the market. "GrafGuard 280-50N offers customers a much wider range of polymer systems such as polypropylene, polystyrene, PET, nylon, and ABS, than our previous products," says Jeff Gough,

IMAGE: NEOGRAF SOLUTIONS



Above: NeoGraf Solutions says GrafGuard 280-50N expandable graphite flake additive is widely used as a flame retardant in thermoformed and extruded thermoplastic panels used in the aerospace and automotive industries

Product Manager for NeoGraf. The company recommends using MgOH as a synergist.

Radical alternatives

New FR developments at the **Fraunhofer Institute LBF** for Structural Durability and System Reliability focus on the extension of the oxyimide radical generator family. Previous oxyimide flame retardants comprising ester linkages influence processability of engineering plastics. The new family of oxyimide ethers is said to be compatible with polyamides and can provide UL 94 V-0 in PA6 at concentrations as low as 3%. Flame retardancy is provided through the formation of a glassy layer as a barrier, which Prof Dr Rudolf Pfaendner, Division Director Plastics at the institute, says seems to be a mechanism not seen before.

Another advanced research topic at LBF is the design of flame retardants based on renewable resources. These novel molecules combine active phosphorus species as part of a polymer chain synthesised using readily available raw materials such as cellulose, lignin, and pentaerythritol. One application area is to provide flame retardancy for biopolymers, most notably PLA. Adding 5% of flame retardant to PLA results in UL 94 V-0, whereas with a commercial polyester flame retardant a 15% loading achieves only V-2 due to burning dripping.

Synergist developments

Paxymer, which developed its own synergist for flame retardants, says it has been involved in several exciting developments during the year. "Due to improvements in the production of the synergist there has been a significant improvement in efficiency of the synergist itself," says CEO Amit Paul. "It improves residue levels in materials by more than 250% by adding 1-2 wt% of synergist in combination with conventional P/N systems."

The main benefits come from reducing the flame spread, eliminating dripping, and increasing gas phase availability of the P/N systems, enabling compounders to meet fire standards with a lower total level of flame retardant. "This often means cost savings - sometimes up to 20% on HFFR formulations," says Paul. Paxymer's latest development has increased the thermal stability so that that the synergist can be processed at up to 300°C. It is now also said to be easier to handle and less sensitive to moisture.

Preliminary trials have proven compatibility and efficiency in polyamides and ABS. "We were previously focused only on polyolefin plastics, but the new product is compatible more or less across resins," says Paul. The company has also developed new masterbatch formulations that are aimed at conduit and duct markets in PP and PE. Addition levels below 10% can achieve self-extinguishing performance.

Bansi L Kaul, CEO of **MCA Technologies**, says applicability of the company's morpholino-poly(piperazinyl-morpholinyl-triazine) proprietary universal fire retardant synergist and smoke and toxic gases suppressant has been extended beyond its original purposes. "It is now found that MCA PPM Triazine HF additionally acts as a heat stabiliser, beyond anti-oxidants, to retard visible and invisible degradation during melt-processing and reprocessing (recycling) of invariably high-loaded (and heat sensitive) FR compounds, such as PINFRs," he says.

Composed of C, H, N elements and characteristically insoluble, MCA PPM Triazine does not bloom or bleed during the service life of the plastics compound. "No matter in what polymer we tested we always found the stabilising effect," says Kaul. "PPM Triazine HF has also shown to be a facilitator for environmentally safer ultimate disposal of waste plastics, beyond recycling, thereby enabling energy extraction with least production of toxic gases." ❯

Table 2: Performance of novel polymeric bio-based flame retardants in PLA

PLA grade	Flame retardant	UL 94 V
low viscosity	no	V-2
low viscosity	15 % Polyester	V-2
low viscosity	5 % LBF-Polymer	V-0
high viscosity	no	Not classified
high viscosity	15 % Polyester	V-2
high viscosity	10 % LBF-Polymer	V-0

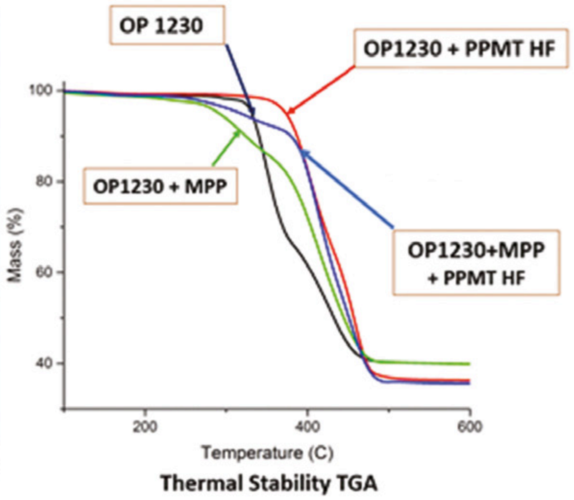
Source: R Pfaendner, Fraunhofer Institute

Figure 5: Table and graph demonstrating how MCA PPM Triazine improves thermal stability (TGA) and fire retardancy of 35% glass reinforced PA66 (Durethan AKV35CXH2.0 from Lanxess)

Source, MCA Technologies

Composition	TGA °C**	LOI	UL 94
GF PA 66 (LANXESS)	N.C.	24.3	N.C
+ 20% OP1230 (Clariant)	331	48	V-1
+ 12% OP 1230 + 8% HF (MCAT)	373	37.3	V-1
+ 12% OP 1230 + 8% MPP (Budenheim)	291	28.3	V-0
+12% OP 1230 + 4% HF + 4% MPP	316	30.7	V-0

(** Onset of weight loss)



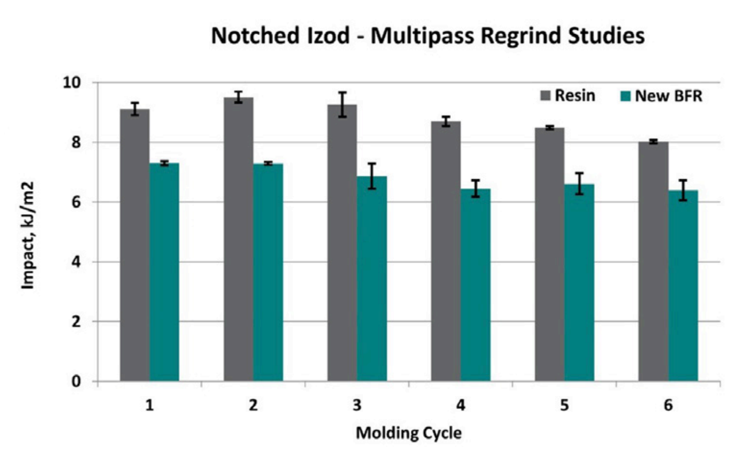


Figure 6: Notched Izod impact property retention of HIPS and HIPS compounds containing Saytex Alero polymeric FR after multiple recycling passes
Source: Albemarle

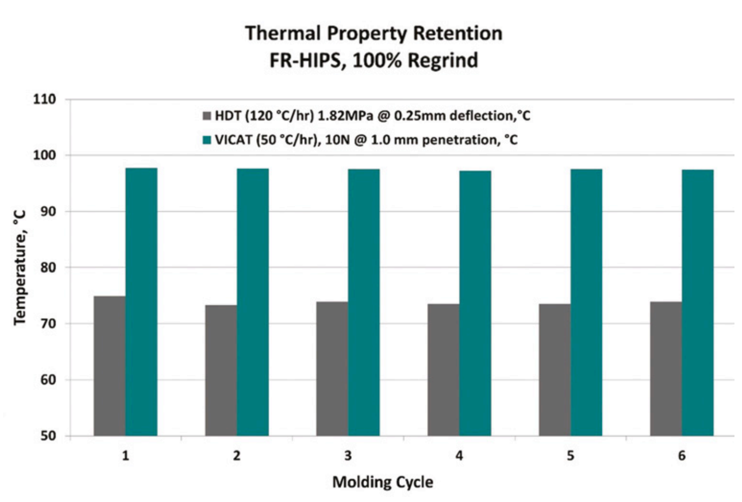


Figure 7: Thermal property retention study – HDT and Vicat – for Saytex Alero in HIPS after multiple recycling passes
Source: Albemarle

Backing bromine

Suppliers of bromine-based flame retardants (BFRs), which are still used in vast quantities in plastics and other materials, are holding their ground. Matt Von Holle, Vice President Global Business Leader Fire Safety Solutions at **Albemarle Corporation**, strongly defends bromine-based chemistry. Albemarle's diverse product offerings include large, brominated molecules in its Saytex branded portfolio, including 8010 ethylene-bis(pentabromophenyl), or EBP, and BT-93W ethylene-bis(tetrabromophthalimide), or EBTBP, both of which Von Holle says have excellent toxicology profiles.

"These molecules are non-bioaccumulative due to insolubility in water and organic media, including fatty oils," he claims. "Compounds of these molecules have also been tested in mechanical recycling studies, showing excellent retention of flammability, physical, mechanical, rheological, and thermal properties, oftentimes better than the base resin itself."

Von Holle goes on to say that a study in flame retarded high impact polystyrene (FR-HIPS) containing EBP mimicking post-industrial recycling (PIR) operations at the high end of processing temperatures (250°C) at 10% regrind level, found no observable change in EBP concentration via liquid chromatographic (LC) analysis after six moulding cycles. "Further, independent laboratory studies have confirmed no presence of dioxin or dibenzofuran in the EBP flame retardant raw material, nor in any thermoplastic formulation before or after mechanical recycling," he says.

Albemarle is continuing to develop what Von Holle calls new sustainable and high-performing brominated flame retardants. At flame retardant conferences organised by *Compounding World* publisher AMI in 2019, the company introduced a

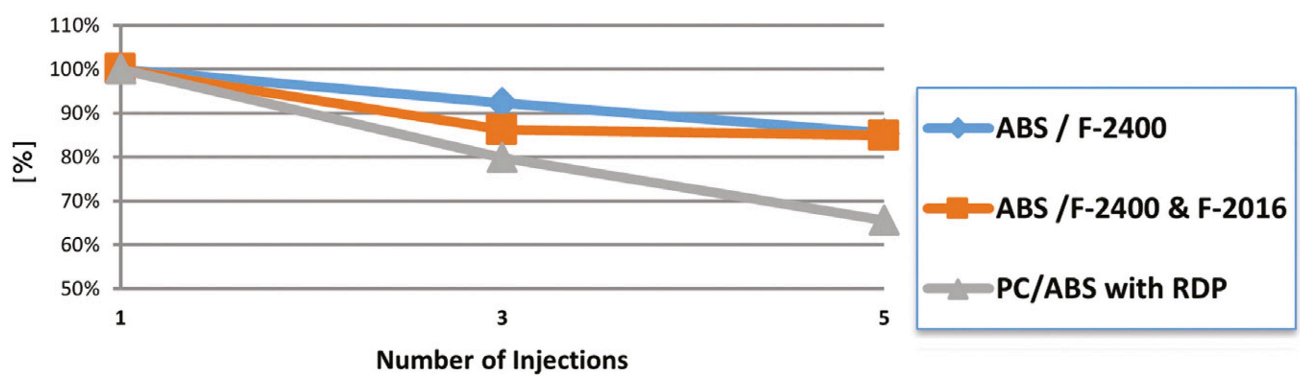


Figure 8: Change in Izod impact strength of different flame retardant ABS and ABS/PC formulations after recycling
Source: ICL

new polymeric FR – Saytex Alero – showing its thermal stability and suitability for recycling. It is said to exhibit improved performance characteristics in HIPS and ABS, including heat resistance and impact resistance, which were shown in an injection moulding study to be retained after multiple passes (Figures 6 and 7).

Recycling issues

At **ICL**, a major supplier of brominated and non-brominated flame retardants, Technical Marketing Support Manager Marc Leifer, together with colleagues Eyal Eden and Lein Tange, has also been looking at recyclability of FR compounds and how suitable different types are for electronic displays.

ICL reviewed typical compounds widely used in the manufacture of electronic displays. It put the compounds through five cycles of injection moulding and mechanical recycling. The first cycle involved virgin compound, while cycles two to five were performed by mixing 50% of the initial compound with 50% of recycled material from the previous cycle. Tests were performed at each stage on flammability, mechanical and rheological properties.

Three UL94 V-0 formulations were tested: two were based on ABS, containing respectively ICL's F-2400 polymeric high molecular weight epoxy brominated fire retardant (BFR) and ATO; and a blend of F-2400 with its F-2016 epoxy BFR and ATO. The third compound was based on PC/ABS (70/30), using ICL's Fyrolflex RDP as the flame retardant.

UL94 V-0 values were maintained throughout along all five cycles for the ABS/BFR formulations. The PC/ABS formulation maintained UL94 V-0 for the first cycle only and failed at cycles 3 and 5. Both ABS/BFR formulations exhibited an Izod Impact reduction of 15% through the fifth cycle (Figure 8). A 34% reduction was recorded for the PC/ABS/RDP formulation. ABS formulations showed a 23% increase in MFI (220°C/10kg). MFI for the PC/ABS formulation increased by over 200% (250°C/5 kg).

As far as resistance to heat and humidity were concerned, injection moulded specimens were aged under the following conditions: oven temperature 80°C; relative humidity 95%; ageing period of 168 hours. The ABS/BFR formulations maintained UL-94 V0, while the PC/ABS/RDP ended up with a rating of V1. The ABS formulations showed a decrease of 13-17% in Izod impact strength, while in the PC/ABS formulation it fell by 32%.

ICL also assessed the greenhouse gas emissions (CO_2 equivalents) of several flame-retarded TV housing formulations throughout the lifecycle of the products. The results showed that HIPS and ABS containing brominated FRs exhibit a lower carbon footprint (12.9 and 16.5kg respectively per housing) than a PC/ABS containing BDP (21.7kg).

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