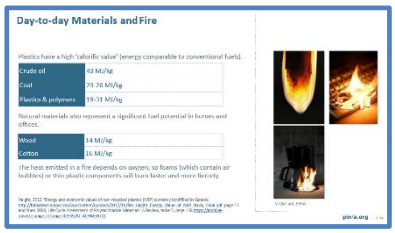
pinfa
Phosphorus, Inorganic & Nitrogen Flame Retardants Association

Your newsletter for non-halogen fire safety solutions n° 125 April 2021

PINFA



New tool to teach fire safety and FRs

80+ slides of information and graphics explaining materials fire safety, flame retardants, fire tests, standards, environment and health. This new toolkit for teaching, professional training and industry, is made available for public use on request by pinfa. It has been developed with expertise from the nearly forty member companies of pinfa in Europe and worldwide (with pinfa North America and pinfa China) as well as external experts. Sections of the slide set cover materials fire safety, how flame retardants work to prevent, extinguish or slow fire development, fire tests and standards, regulations, environment & health questions, flame retardants markets and applications, including wire & cable, building (construction), transport. pinfa intends to extend the slide set content in 2021 to cover recycling, bio-based FRs and other applications (E&E, textiles, wood/timber, e-mobility, 3D printing).

Download the slide set in PDF, Powerpoint version available on request: https://www.pinfa.eu/media-events/pinfa-academy/

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REGULATORY

US EPA consultation on Deca, HBCD, TIPPP

Public comment is requested on rules to reduce exposure to PBT chemicals including three FRs: Deca-BDE, HBCD, TIPPP. Deadline for input is 17th May 2021. Deca-BDE is prohibited in the USA from January 2022 (sale of articles to which Deca-BDE has been added), with longer deadlines for some applications and an exception for recycling of Deca-BDE containing plastics. TIPPP (Tris (4-isopropylphenyl) phosphate) and its use were banned in January 2021, with exceptions in some uses. HBCD and its use were banned in January 2021, with no use exceptions other than disposal as waste fuel. The EPA requests input to justify possible exceptions to these bans, conform to the US regulatory obligation (TCSA) to implement bans of such PBT (persistent bioaccumulative toxic) chemicals "as soon as practicable".

"EPA requests comment on PBT chemicals", ***deadline for input 17th May 2021*** *https://www.safetyandhealthmagazine.com/articles/21038-epa-requests-comment-on-pbt-chemicals*

Unlimited fines for fire safety regulations

The UK Government has announced unlimited fines for breaching fire safety orders or obstructing fire inspectors. This is part of a package to tighten fire safety in buildings following the Grenfell fire and including: improving fire safety coordination and clarifying responsibilities, keeping fire safety information over the whole building lifespan, improving fire safety risk assessments, strengthening guidance under Fire Safety Orders to enable failure to respect it to lead to legal proceedings, overall strengthening of enforcement, and increased funding for fire and rescue services.

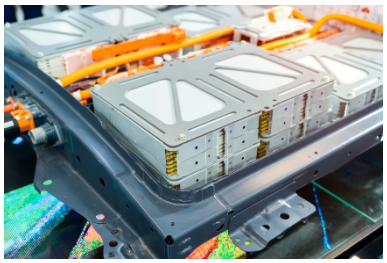
"Unlimited fines for breaches to fire safety regulations", 18th March 2021 https://www.fmj.co.uk/unlimited-fines-for-breaches-to-fire-safety-regulations/

Sustainable Products Initiative consultation

EU public consultation to 9th June 2021 on the Sustainable Products Initiative, targeting mainly recycling and reuse. This Initiative stems from the Green Deal and the new Circular Economy Action Plan and aims to promote products which have low climate impact, are resource efficient, reduce waste and enable circularity. Amongst proposed measures under 2A Design for sustainability are requiring information on how products are "safe-by-design" and have substituted chemicals of concern, requiring information on chemicals or banning the use of chemicals which inhibit recyclability. Two of the proposals under 2B Digital Product Passport also address chemicals in products.

Sustainable Products Initiative – EU general public consultation. Open to 9th June 2021 https://ec.europa.eu/info/law/better-regulation/have-your-say/initiatives/12567-Sustainable-Products-Initiative/public-consultation

BATTERIES



Review on flame retarding of batteries

Fire and thermal runaway risks of lithium ion batteries can be reduced by using PIN FRs in separators, electrolyte, cathode. A review of materials for improving thermal stability and safety of lithium ion batteries (LIBs) provides information and references on different PIN FR solutions in current battery technology and possible future battery designs to improve safety. LIB fires are a significant problem, and incidents are cited in aircraft, smartphones, energy storage systems and electric scooters. Flame retardants are relevant for battery casings and structure (not discussed in this paper), for flammable battery coolants, in battery electrolytes (currently organic solvents), separators (polymer based) and incorporated into the anode or cathode.

Conventional LIB electrolytes are usually based on lithium hexafluorophosphate dissolved in solvents. FRs used in these electrolytes include inorganics (e.g. silica based) and “organic phosphorous compounds, with various types, low toxicity, suitable physical characteristics, good compatibility and low cost” (10+ references) or fluorinated carbonates. A future development to reduce fire risk may be solid-state electrolytes, e.g. ceramics or polymer-based gels, or ionic liquids or gels.

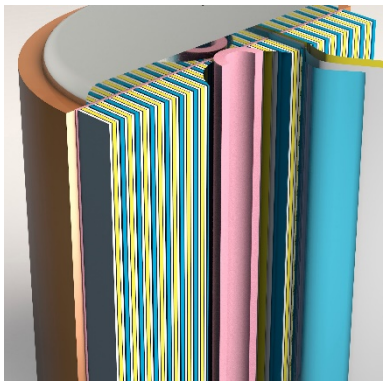
LIB separators are a critical element of battery cells, and if compromised can allow short circuit and thermal runaway. Conventional separators are polyolefin-based membranes. Mineral PIN materials (e.g. based on silica or aluminium) and ceramic coatings can improve separator safety.

LIB cathodes are based on various lithium compounds, play an active role in the electrical process and contribute to fire risk. Thermal stability of cathodes and safety can be improved by incorporating or coating with PIN materials such as aluminium phosphate, nickel, magnesium or other mineral compounds.

Risks related to LIB anodes particularly concern lithium dendrite formation and can be reduced by mineral doping or coating.

“A Review on Materials for Flame Retarding and Improving the Thermal Stability of Lithium Ion Batteries”, F. Gao et al., Int. J. Electrochem. Sci., 15 (2020) 1391 – 1411, https://dx.doi.org/10.20964/2020.02.24

See also the detailed review of lithium ion battery fire risks, impacts and fire prevention “A review of lithium ion battery failure mechanisms and fire prevention strategies”, Q. Wang et al., Progress in Energy and Combustion Science 73 (2019) 95–131 https://doi.org/10.1016/j.pecs.2019.03.002



Review on fire safety of battery separators

Flame retardant separators are important to prevent or limit lithium ion battery fires and thermal runaway. The separator in lithium ion battery cells prevents direct contact between anode and cathode, leading to internal short circuit, resulting in overheating, and then decomposition of anode material to release oxygen which can react with the electrolyte and the separator. Conventional separators are based on polyethylene and polypropylene, which can shrink under heat (leaving holes) or burn. Strategies to address this battery safety problem include use of higher performance polymers, incorporation of minerals into the polymer (titanium, aluminium, zirconium, silica), ceramic-polymer multilayer separators and use of flame retardant additives in the separators (e.g. ATH, MDH, organic and inorganic phosphates, melamine compounds …). The problem would be reduced with solid state electrolytes but to date these show low electrical performance and often high sensitivity to moisture or oxygen.

"Recent progress in flame-retardant separators for safe lithium-ion batteries", X. Zhang et al., Energy Storage Materials 37 (2021) 628–647 https://doi.org/10.1016/j.ensm.2021.02.042

Full-scale fire test of EV battery

In just 22 seconds, cell thermal runaway spreads flames throughout the battery compartment. A full-scale fire test was carried out on a battery system of seventeen 3P6S battery modules mounted with control systems in a car chassis. One battery module was overcharged until thermal runaway occurred. Within five seconds, thermal runaway spread to the four adjacent modules. Released gas was immediately ignited, with jet flame and smoke, and temperatures reached over 600°C. These five modules then smouldered, and further modules ignited after around two minutes. The authors note that water fire suppression would be hindered by the battery pack casings.

"Full-Scale Experimental Study on the Combustion Behavior of Lithium Ion Battery Pack Used for Electric Vehicle", H. Li et al., Fire Technology, 56, 2545–2564, 2020 https://doi.org/10.1007/s10694-020-00988-w

Efectis report on battery fires

Report to EU underlines fire safety problems of lithium ion batteries, incidents in uses from electronics to airplanes. A summary report by Efectis for the European Commission's Fire Information Exchange Platform summarises fire risks of lithium ion batteries in manufacturing, battery transport and storage and in uses including electric vehicles, e-bikes, aircraft, energy storage and consumer electronics. The report concludes that fire safety is an issue with lithium ion batteries, often related to design defects or damage during handling or use, leading to short circuits and then thermal runaway.

Many questions remain unanswered concerning fire interventions, especially for e-vehicles in car parks and energy storage systems, e.g. linked to photovoltaics.

"Technical report on battery fires", Efectis, for the Fire Information Exchange Platform, 14th May 2020, presented to the FIEP webinar of 4th March 2021

PRODUCT INNOVATION

PIN FR polyamide PA6 for 3D-printing

BASF FORWARD AM offers a non-halogenated FR PA6 for 3D-printing of performance parts for public transport and electronics. The PA6 compound power for Powder Bed Fusion 3D-printing is the first such material to be UL Blue Card certified, passes ECE-R 118 App 7 (public transport) and achieves UL94-V2 (0.8 mm), GWI glow wire ignition 750°C @ 1 mm, smoke density BSS 7238 Rev C, gas toxicity BSS 7239 Rev A and FST smoke density and toxicity for aerospace applications. Mechanical properties include high rigidity, thermal resistance and good tensile strength. Applications include switchboard and electronic components, air ducts for aerospace, fixtures for cables and pipes.

"Ultrasint® PA6 FR. Halogen-Free Flame-Retardant PA6 Powder for Advanced Applications", https://forward-am.com/material-portfolio/ultrasint-powders-for-powder-bed-fusion-pbf/pa6-line/ultrasint-pa6-fr/ and "Enabling Flame Retardant Applications with Ultrasint® PA6 FR", BASF FORWARD AM, December 2020 https://forward-am.com/use-cases-and-whitepapers/enabling-flame-retardant-applications-with-ultrasint-pa6-fr/

Graphite PIN FR for railways

Tirupti Graphite has a first order for non-halogenated FR polyurethane foam and composite for railway carriages. Expandable graphite provides PIN fire protection to achieve European railway fire safety standard EN 45545 Hazard Level 3 (highest) for polyurethane foam for seating and berths. Indian Railways have contracted supply for an initial 50 railway carriages. The Tirupati products are based on natural flake graphite with up to 96% purity and are REACH registered in Europe. Grades are available for use in polyurethane foams, rubber and latex foams

"Tirupati Graphite starts European sales of expandable graphite after certification win", 10th February 2021 https://www.proactiveinvestors.com/companies/news/940917/tirupati-graphite-starts-european-sales-of-expandable-graphite-after-certification-win-940917.html and "Tirupati Graphite wins first order for new fire-retardant foam from railway carriage maker in India", 17 December 2020 https://www.proactiveinvestors.co.uk/companies/news/936832/tirupati-graphite-wins-first-order-for-new-fire-retardant-foam-from-railway-carriage-maker-in-india-936832.html

Developing PIN FR for expanded foam parts

Unipetrol is working on low-migration, halogen- and antimony-free fire safety in lightweight automotive foamed parts. Expanded foamed polymer parts (e.g. polypropylene) combine reduced materials consumption, weight savings and mechanical performance but pose flammability challenges. Unipetrol (Orlen Unipetrol Group, Czech Republic) considers that current halogenated and antimony FR solutions will decline as manufacturers push for PIN FR solutions with preferable environmental and toxicity profiles. A combination of synergist PIN additives has been tested which achieves fire performance HF-1 and VTM-0 (80 – 100 mm/min), with low migration and good foaming and film stretching compatibility at loadings up to 30%. Further work is underway to reduce flammable drips.

"Halogen-free flame retardants for foamed PP parts and film", Compounding World, December 2020
http://download.polympart.ir/polympart/EMag/CW-Dec-2020.pdf

Thermally conductive epoxy resins for LEDs

PIN FRs enable fire performance, low smoke and fumes, transparency and thermal conductivity for LEDs. Electrolube, a global formulated chemical supplier since 1941 to electronics and industry, has developed innovative thermally conductive flame retardant epoxy resins. The white ER2074 resin uses PIN flame retardants to achieve UL94-V0) with low smoke and fumes. The resin is thermally conductive (1.26 W/m.K), does not contain abrasive fillers so is processing friendly, and offers high water and chemical resistance. Its white colour after curing offers appearance advantages and combined with its thermal conductivity makes it ideal for applications in LED lighting, both internal and external. Electrolube has now announced a first transparent UL94V-0) resin, UR5641, again using PIN flame retardants, and using aliphatic urethane chemistry resistant to yellowing. It offers thermal conductivity of 0.35 W/m.K and operating temperatures -40 to 120°C.

https://electrolube.com/product/er2074-thermally-conductive-white-epoxy-resin/

"Electrolube Launches First Clear UL94 V-0 Flame Retardant PU Resin for any LED Market" 23 October 2019
https://electronicsmags.com/electrolube-launches-first-clear-ul94-v-0-flame-retardant-pu-resin-for-any-led-market/ and
https://www.electrolube.info/pdf/tds/044/UR5641.pdf

Rubber flooring for rail fire safety

Treadmaster PIN FR flooring offers fire safety beyond standard rail requirements, wear resistance and visual appeal. Tiflex is a leading producer of cork and bonded rubber materials for over 70 years. Their Treadmaster TM7 synthetic rubber flooring is homogenous, ensuring consistent appearance and optimal integrity under heavy wear, and enabling repair in case of local damage. It is slip-resistant and can be supplied to individual colours, designs and specifications. The material uses non-halogenated flame retardants to achieve not only the European rail standard EN 45545-2 HL3 on all substrates, but also BS69853:1999 cat. 1a which can be more stringent. The flooring can integrate photoluminescent materials to provide emergency lighting, and this is already adopted by Bombardier and QNGR

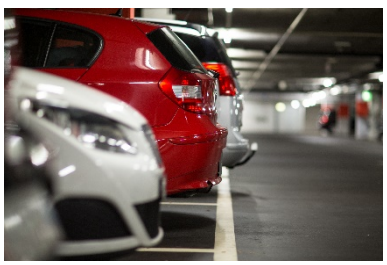
"Tiflex Brand Treadmaster Flooring Excels in Fire Safety", 2 June 2020 https://railway-news.com/tiflex-brand-treadmaster-flooring-excels-in-fire-safety/ and Tiflex website http://www.treadmasterflooring.com/index.html

Performance FR cables for solar PV

Cicoil low-smoke, zero-halogen FR cables for reliable, weather and UV resistant performance in solar and technical uses. The Cicoil Flexx-Sil jacketed single and multi-core cables are designed for up to 42 KV (DC) and do not require protection in harsh environments including sunlight, extreme temperatures (-65 - +260 °C), mechanical stress, acid rain, salt, humidity. The cables can be supplied transparent, and do not deform or become brittle with ageing. The outer jacket is self-healing from small punctures. The PIN (non-halogenated) flame retardant cables pass UL/CSA CW-1, FT1 & FT2, UL94-V0 and FAA fire tests and are low offgas to ASTM E595. Cicoil are producing performance cables since 1956, with experience including equipping the first space walk and the lunar landings of 1965 and 1969.

"Ultra-Durable Flexx-Sil™ Jacketed Solar Panel Power Cables", 8 December 2020 https://www.cicoil.com/news/product-news/item/ultra-durable-flexx-sil-jacketed-solar-panel-power-cables

RESEARCH

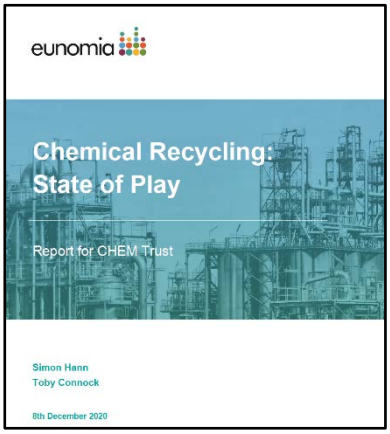


Increasing fire risk of car parks in buildings

Study concludes that increasing fire hazard of vehicles means more "devastating fires" in parking structures. In a 30 page analysis based on nearly eighty references, the authors conclude that vehicle parking structures represent an increasing risk of major fires because of larger vehicles, increasing use of polymers and other combustible materials in vehicles, plastic fuel tanks, use of

plastics in bodywork (facilitating fire spread to other vehicles), electric vehicles, because of changes in parking design, such as multi-level garages, denser parking slots and also mechanical vehicle stacking/parking systems, and also because of a general lack of requirements for active fire protection systems in parking in fire codes. Weight of plastics in cars is estimated to have doubled from c. 80 kg in the 1970's to 160 kg today. The authors conclude that fire codes and regulations should not be based on fire test data for vehicles older than 2000-2005.

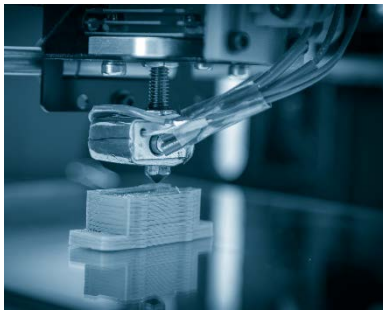
"Fire Hazard Analysis of Modern Vehicles in Parking Facilities", H. Bohmer et al., Fire Technology 2021 https://doi.org/10.1007/s10694-021-01113-1



NGO report on chemical plastics recycling

Environment NGO recognises potential of chemical plastics recycling but considers more LCA and energy data is needed. A report by Eunomia for the environmental NGO CHEMTrust assesses solvent purification, chemical depolymerisation and thermal depolymerisation (cracking / pyrolysis), concluding that chemical depolymerisations appear to have most promise (e.g. for PET, polyester), but should not replace mechanical recycling where clean, homogenous material can be collected. The report however considers that to date inadequate information is available on chemical recycling processes: environmental and energy performance, losses in recycling, extent to which sorted and clean input streams are needed, by-product / waste streams. The report recognises that the lack of information is because the technologies are mainly at the development or pilot scale, and companies wish to keep process information confidential for commercial reasons.

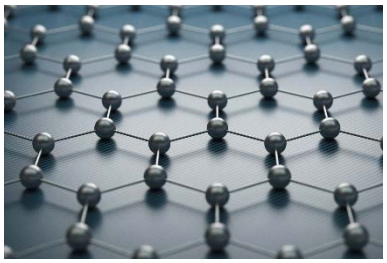
"Chemical Recyling: State of Play", report for CHEM Trust, S. Hann, T. Connock, 8th December 2020 https://chemtrust.org/wp-content/uploads/Chemical-Recycling-Eunomia.pdf



Flame retardants and 3D printing

Review points to PIN FR 3D-pinting materials but suggests more data is needed on fire behaviour of 3D-printed materials. A review of nearly 80 sources summarises fire behaviour of 3D-print polymers and developments of PIN FRs. Poly-lactic acid (PLA), ethylene-vinyl acetate (EVA), polyetherimide (PEI, polyamide and silica aerogels are reviewed, with various PIN FRs. Commercial PIN FR 3D-printing filaments CRP Windform www.windform.com and Markforget Onyx https://markforged.com/materials/plastics/onyx-fr are cited, both achieving demanding aviation fire safety standards. The review notes that 3D-printing can lead to significant differences in flammability compared to other polymer processing systems, because of fill density and layer orientation, and that further science research is needed.

"Fire Behavior of 3D-Printed Polymeric Composites", K. Babu et al., Journal of Materials Engineering and Performance JMEPEG, March 2021 https://doi.org/10.1007/s11665-021-05627-1



Graphene effective PIN FR for foams

PET foam treated with Directa Plus graphene resists flame penetration seven times longer than neat foam. The tests were carried out by Turin Polytechnic using 120 kg/m^3 polyethylene terephthalate foams from BASF, coated with Directa Plus expanded graphene nanoplatelets. The graphene was deposited from water based solutions, using three different dispersants, by manual brush painting onto the foam sample surface (approx. 1.5 – 3.5 % w/w coating/foam). UL94 horizontal and vertical burning tests were carried out using 20 mm thickness foam samples, and flame penetration test using 2 mm thickness samples. The coating interrupted horizontal and vertical burn, increased time to ignition, reduced peak heat release rate by up to 60% and increased time to flame penetration by seven times.

"Directa Plus says new research demonstrates graphene's flame retardant properties"
https://www.proactiveinvestors.co.uk/companies/news/940895/directa-plus-says-new-research-demonstrates-graphene-s-flame-retardant-properties-940895.html

"PET Foams Surface Treated with Graphene Nanoplatelets: Evaluation of Thermal Resistance and Flame Retardancy", S. Matta et al., Polymers 2021, 13, 501, https://doi.org/10.3390/polym13040501

Mineral PIN FR cables show durability

Cables with mineral PIN flame retardants showed durable fire performance after prolonged harsh weathering. Simple test cables (copper core plus mono-material sheath) were formulated by Leoni Kabel, using EVA and EVA-LLDPE (ethylene vinyl acetate and linear low density polyethylene) and the well-recognised mineral PIN flame retardants ATH and MDH (aluminium tri hydroxide at 35-40% loading and magnesium di hydrate at 60% loading, both supplied by Nabaltec), plus 0.25% antioxidant. The cables were exposed to cycles of temperature (-10°C to +70°C), UV and humidity in a weathering chamber for up to 12 weeks (continuous cycles). Fire performance of the cables was little changed after weathering (e.g. peak heat release rate decreased slightly). After 12 weeks weathering, serious structural problems showed with all cables, with visible cracks and degradation of sheaths. The authors conclude that there will be no problem with flame retardancy performance for such materials subjected to natural weather exposure for several years, but that durability of fire retardancy for longer periods requires further investigation.

"Degradation of flame retardance: A comparison of ethylene-vinyl acetate and low-density polyethylene cables with two different metal hydroxides", Y. Tan et al., J Appl Polym Sci. 2021, https://doi.org/10.1002/app.50149

FRs improve wood product fire safety

Tests show that surface-applied intumescent FRs prevent or delay ignition of wood products and reduce heat release. Three FR formulations were compared in cone calorimeter fire tests to virgin products (no FR), including MDF (medium density fibreboard), blockboard and pine. The FRs tested were intumescent, or intumescent plus phosphate ester. Heat fluxes of 35 – 80 kW/m^2 were applied and time to ignition, peak heat release rate, average heat release rate, effective heat of combustion and gas toxicity (FTIR) were tested. The FRs resulted in either no ignition (including in all samples at 35 kW/m^2) or considerably increased ignition time (2 -32x longer time). Peak heat release rate was reduced by 2 – 5 times. Toxic emissions were reduced when FRs prevented ignition (except for ammonia emissions from the intumescent) but were increased in cases of ignition, with carbon monoxide more than doubled in some cases, acrolein increased by 10-60 % and increased HCN.

"Thermal behavior and toxic emissions of flame retarded timber in cone calorimeter tests", D. Tsatsoulas, Int. J. of Safety and Security Eng., Vol. 1, No. 1 (2011) 45–64 https://dx.doi.org/10.2495/SAFE-V1-N1-45-64

OTHER NEWS

Endocrine (EDC) effects of "replacement" FRs. Experimental data and endocrine modelling results were collected for 52 flame retardants considered to be possible replacement of brominated PBDE and HBCD FRs. Experimental data was found for less than half of the FRs (23 / 52). Of these: nine showed no EDC activity (EHDPP 2-ethylhexyl diphenyl phosphate, 2,4,6-tribromophenol, TEHP Tris(2-ethylhexyl) phosphate, TCEP Tris-2-chloroethyl phosphate, TEP Triethyl phosphate, DBPNG Dibromoneopentylglycol, PBEB Pentabromoethylbenzene, HBB Hexabromobenzene, TnPP Tri-n-propyl-phosphate) and a further eight showed "Weak evidence" of activity. Only seven FRs showed "Godo" or "Strong" evidence of EDC. Model predictions appear to have little correlation to experimental results for EDC. The authors conclude the need for further endocrine effect testing of FRs, possible future regulation and possible concerns about effects of combined exposure to mixtures of a number of FRs and other chemicals.

Developmental effects of brominated FRs. A mixture of tetra- and deca-BDE and HBCDD was fed to female rats during pregnancy and lactation. At a dose of 0.06 mg/kg body weight/day, estimated to be comparable to maximum human intake (ingestion of 100 mg/day of dust), resulted in significant premature development of mammary glands in female offspring.

Two triaryl P-ester FRs show behavioural impacts on zebrafish. Tris-cresyl phophate and cresyl diphenyl phosphate were tested at 5 and 25 µl/l (that is parts per billion), with c. 0.5% dimethyl sulfoxide as a cosolvent, on adult zebrafish for 15 days. The pesticides cypermethrin and methomyl were tested similarly (these molecules do not contain phosphorus, but respectively chlorine and sulphur). Swimming strenth and swimming behaviour were affected with all of the chemicals, with higher impact at the higher dosage.

Replacing old fire-safe furniture reduces levels of FRs in household dust. FRs were analysed in household dust in 42 homes, before and (up to 18 months) after replacing fire-safety treated sofas (pre-2014 California TB117, that is furniture dating from 1975 to 2014) by non-FR furniture. Dust was collected on and around the furniture. Three brominated PBDEs, three chlorinated PFRs and one non-halogenated P-ester (triphenyl phosphate) were found in most homes and two non-halogenated P-FRs were not detected (TNBP, TPP). The detected FRs all showed lower levels in dust in nearly all homes after furniture replacement. pinfa notes that replacement with new FR furniture was not studied, so that it cannot be stated whether the lower FR levels in dust are due to changing to non-FR furniture or simply due to replacement of old furniture.

"Synthetic organic chemicals (flame retardants and pesticides) with neurotoxic potential induced behavioral impairment on zebrafish (Danio rerio): a non-invasive", Z. Ren et al., Environ. Sci. Pollut. Res. 2021 https://doi.org/10.1007/s11356-021-13370-2

"In Utero and Lactational Exposure to an Environmentally Relevant Mixture of Brominated Flame Retardants Induces a Premature Development of the Mammary Glands", R-J Gouesse et al., Tox. Sciences, 179(2), 2021, 206–219 https://dx.doi.org/10.1093/toxsci/kfaa176

"Endocrine disrupting potential of replacement flame retardants – Review of current knowledge for nuclear receptors associated with reproductive outcomes", L. Bajard et al., Environment International 153 (2021) 106550 https://doi.org/10.1016/j.envint.2021.106550

"Do flame retardant concentrations change in dust after older upholstered furniture is replaced?", K. Rodgers et al., Environment International 153 (2021) 106513 https://doi.org/10.1016/j.envint.2021.106513

PUBLISHER INFORMATION

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For abbreviations see: www.pinfa.org