pinfa
Phosphorus, Inorganic & Nitrogen Flame Retardants Association

Your newsletter for non-halogen fire safety solutions
No. 79 May 2017

PIN flame retardants (FRs) are at the centre of innovation in polymer technologies, driven by objectives of fire safety (including low smoke) and sustainability, alongside polymer performance, quality and reliability. The pinfa Newsletter presents a selection of news concerning PIN FR research and applications, with both science publications and industry developments. Innovation involves flame retardant producers, compounders and polymer companies, through to final consumer or industry product designers and manufacturers. This issue covers a range of PIN FR fire safety solution ingredients (zirconium, titanate, glass microspheres, phosphorus, nitrogen, clay minerals) and innovations addressing sustainability (bio-waste sourced PIN FR chemicals, sustainable chemistry, hazard and exposure assessment, recycling and the circular economy).

Upcoming events

26-28 Sept., Dearborn, MI, near Detroit, USA, **pinfa NA 5th annual workshop**: **Fire Safety Requirements in Automotive Design**. This workshop, co-organised with SAMPE (Society for the Advancement of Material and Process Engineering) will assess how trends in automobile design will impact flame retardant selections and will include car manufacturers and suppliers, regulatory and materials technologies experts. **Includes optional tour of Ford Motor Company Rouge Plant where Ford's F-150 trucks are assembled**
http://www.pinfa.org/images/events/AUTOMOTIVE-CONF-FLYER_04-24.pdf

For full events listing, see www.pinfa.eu

Pinfa – TMP Italy workshop on E&E polymers

Milan, 23rd February 2017. The joint workshop organised by the Italian Technical Plastics Materials industry association (TMP) and pinfa brought together ca. 80 fire fighters, E&E manufacturers, fire safety and regulatory experts and polymer compounders. The workshop enabled dialogue between technical polymer users and the PIN flame retardant industry, addressing trends in industry and regulatory requirements and PIN fire safety solutions.

Loredana Faccincani, **TMP** co-opened the meeting for TMP, underlining the importance ensuring fire safety of E&E equipment, to prevent electrical faults or

pinfa, Avenue E. Van Nieuwenhuyse 4, 1160 Brussels, Belgium
pinfa@cefic.be Phone +32 2 676 74 36 www.pinfa.org
@pinfa_eu

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overloading leading to fires, and the interest of Italy's E&E industry in PIN flame retardants as innovative and sustainable solutions to achieve this.

Philippe Salémis, **pinfa secretary general**, co-opened the meeting, presenting pinfa's vision of continuously improving the environment and health profile of PIN flame retardants, in order to offer sustainable fire safety solutions, as well as a quick overview of pinfa activities (regulatory and fire safety)

Adrian Beard, **pinfa chairman**, and **Clariant**, emphasised the advantages of PIN flame retardants, showing the wide range of different non-halogenated phosphorus, inorganic and nitrogen products today available. PIN FRs can both delay and reduce smoke emission and heat release, compared to some legacy FR systems, so increasing escape time in case of fire. Evaluations by independent studies have already to date (e.g. ENFIRO see pinfa Newsletter n° 36) identified a number of PIN FRs as "generally safe" or of "low level of concern" for health and environment, and have shown that PIN FRs can substitute legacy FRs (e.g. US EPA Alternatives Assessment to DecaBDE, see pinfa Newsletter n° 38). Nonetheless, Work to assess PIN FR environmental and health safety should continue, one example being Clariant's EcoTain® sustainability criteria system (see www.clariant.com/de/Sustainability/Discover-Ecotain).

Paolo Finazzi, **Schneider Electric**, presented the company's requirements for "halogen free" materials. Schneider Electric's has a corporate Planet & Society Barometer policy and operates its own Green Premium ™ Eco Label. Schneider Electric has received a number of awards for environmental and sustainability achievements, including top of the Dow Jones Sustainability Index (DJSI) for the fourth consecutive year in 2016. "Halogen free" is a product requirement for Schneider Electric for three reasons: concerns about release of some halogenated compounds into the environment, fire safety (low smoke, low smoke corrosivity, to improve escape and to avoid infrastructure deterioration) and end-of-life recycling constraints. Consequently, since 2016 Schneider Electric has decided that all new product developments must be "halogen free". Mr Finazzi noted that a number of different definitions and test methods for "halogen free" exist (IEC TC111, TC23 SC23A, 62474 ; DIN VDE V 0604-2-100, DIN VDE 0472 part 815 VDE V 0604-2-100). Schneider Electric currently applies its own internal definition: chlorine + bromine < 0,2% mass and fluorine < 0,1%.

Thomas Futterer, **pinfa vice-chairman** and **Budenheim**, pointed out that the European Commission has launched an EU Circular Economy Action Plan. This will mean higher recycling rates (cradle to cradle), less energy recycling and avoidance of landfill whenever possible (< 10 %). The expected legislative changes may have direct influence on polymer formulations containing flame retardants, bringing questions and tasks to the polymer industry. Thomas Futterer discussed the current challenges of recycling plastics containing flame retardants, resulting from the very wide range of different polymers and of flame retardant combinations which are often tailor-made for specific applications. The pinfa recycling group, consisting of Adeka, BASF, Budenheim, Clariant and Nabaltec is supporting Fraunhofer LBF, which has started a three year technical project to assess the potential for closed-loop recycling of selected polymer-PIN FR systems. Their ongoing study is looking at impacts of multiple extrusion and polymer ageing on fire performance and mechanical properties.

Peter Kulischek, **DuPont Performance Materials**, explained that fire safety performance can create competitive advantage for E&E products. A combination of manufacturing industry sustainability objectives and regulatory constraints is accelerating the move towards non-halogenated flame retardants, but with demanding electrical, mechanical and processing performance requirements. Mr. Kulischek also presented different material property and fire performance requirements for a range of E&E applications, and the tests used to show conformity, covering electrical applications such as automobile, electrical components, motors and relays, circuit breakers. He underlined the accentuation of performance challenges posed by miniaturisation.

Gianluca Mastrodomenico, **IMQ S.p.A**, summarised testing methods relevant to fire performance, heat resistance and tracking for electrical insulation materials. Tests include the Ball Pressure Test (BPT), Determination of the Proof and Comparative Tracking, indices of solid insulating material (PTI and CTI), Needle Flame Test and the Glow Wire Test (GWTEP, GWFI, GWIT).

Jürgen Troitzsch, **Fire and Environment Protection Service** (FEPS) presented developments of regulations and standards applicable to E&E in Europe. He identified as trends in E&E FRs: the decisive importance of environment and health properties, the use of synergistic combinations of phosphorus, nitrogen and inorganic products to achieve fire performance, high molecular weight/polymeric FRs (low toxicity and low bioaccumulation risk, no losses or emissions from polymers). He noted that electrical equipment and cables often also have to conform to regulations and standards applicable in specific applications such as aircraft, shipping, railways or construction. Dr Troitzsch showed the importance of smoke emission, smoke acidity and toxicity requirements in many E&E applications, concluding that in the EU construction products, rail, other transport and cable requirements all consequently drive to move to PIN flame retardant systems.

Hans Wendschlag, **Hewlett Packard**, presented an E&E electrical equipment manufacturer's perspective on developments of flame retardants in Europe and worldwide. The principal ecolabels worldwide restrict halogenated FRs in parts >25g (EU Flower, German Blue Angel, TCO, Nordic Swan, US EPEAT). Most big OEMs offer ecolabel certified products under several of these schemes (although there are no certified products under the EU Flower ecolabel). Some EU member States now authorise ecolabel certification requirement in public procurement. As a consequence, most IT companies now exclude halogenated FRs from all plastic parts and some also now exclude them from printed circuit boards. HP has used a Green Screen™ based assessment process to identify PIN FR alternatives which are preferable.

TMP – pinfa workshop fire safety of E&E polymers and PIN FRs, Milan, 23rd February, 8h30-17h.

Italian Technical Plastics Materials industry association (TMP) and pinfa workshop "Fire safety of E&E polymers and PIN FRs", IMQ, IMQ — Via Quintiliano 43, Milan, 23rd February 2017.

Innovation Spotlight webinar highlights PIN FRs

The Green Chemistry and Commerce Council (GC3) brings together over 100 companies and organisations working on sustainable chemistry across sectors and supply chains. The GC3 webinar of 2nd March 2017 discussed "Greener Flame Retardants", targeting retailers and product end users. The webinar underlined that halogenated flame retardants pose environmental concerns, leading to regulatory restrictions and discussed the supply chain challenges of moving to PIN FRs. Different types of PIN flame retardant were presented, showing the range of chemistries and solutions available. A cross-industry trend to non-halogenated and to polymeric flame retardants was emphasised. Liz Harriman, Massachusetts Toxics Use Reduction Institute (TURI) noted the importance of avoiding "regrettable substitutions" and so the need to carefully assess alternative FRs. Webinar highlighted companies were FRX Polymers, who offer phosphorus-based polymeric flame retardants achieving Green Screen Bench Mark 3 ("use but still opportunity for improvement") and Paxymer, who offer low smoke – no soot PIN FRs and synergists.

Green Chemistry and Commerce Council (GC3) webinar "Greener Flame Retardants". 2nd March 2017 – access slides and watch online at http://greenchemistryandcommerce.org/startup-network under webinars

Titanate for PIN FR cable insulation

A US patent application proposes a combination of a phosphorus flame retardant, silica and organo-titanate compounds to achieve fire performance in thermoplastic polyolefins for data communication cable insulation. The patent application claims that fire and smoke standards NFPA 262, UL 910, UL 1666 and UL 1685 can be achieved. The titanate was used at <1.5% and silica at 2%, and a number of advantages are claimed. With silica, the titanate acts as a synergist, improving the effectiveness of phosphorus and nitrogen PIN FRs (e.g. ammonium polyphosphate, melamine phosphate) and improving char integrity. It also improves dispersion of the PIN FR in the polymer and enhances melt processing. Previous patent documents also claim that the titanate additives prevent melt dripping in fire.

"Halogen-free flame retardant material for data communication cables", US patent application 20170029607 published 2/2/2017 http://www.google.com/patents/US20170029607

Sixteen killed in Hungarian coach fire

Sixteen people, including many children, died and forty were injured when a coach carrying students from Hungary hit a pillar and caught fire near Verona, Italy, on 21st January. A truck driver is reported to have seen black smoke coming from the rear of the Setra S317 GT-HD coach before it collided with a crash barrier, hit a bridge pylon and then caught fire. The fire is thought to have been caused by an electrical short-circuit in lighting poles placed too close to the crash barrier and damaged by the coach, then fuel igniting. Experts have repeatedly called for tighter fire safety regulations in buses and coaches, as these are currently considerably laxer than for railways (see pinfa Newsletter n° 67).

"Italy coach crash: at least 16 dead as bus carrying Hungarian students bursts into flames near Verona", The Telegraph, 22 Jan. 2017 http://www.telegraph.co.uk/news/2017/01/21/least-seven-feared-dead-italian-coach-crash/ and https://en.wikipedia.org/wiki/2017_Verona_bus_crash

Deaths from vehicle fires after road accidents

A report from SP Sweden (now RISE) assesses data on fatalities due to post-collision fires in road vehicles, based on data from Sweden, the USA and the UK. The data suggest that around 5% of road accident deaths are caused by fire in all of these countries. Collisions are the cause of less than 4% of road vehicle fires, with the principle cause being mechanical or electrical failure, but collision fires are responsible for over half of vehicle fire deaths. The report suggests that most of the deaths are caused by smoke inhalation, because smoke incapacitates occupants fast enough to impede escape. Over 30 vehicle fires per hour occur in the USA, causing around 300 deaths and 800 injuries per year. The report states that the large number of fatalities in vehicle fires is related to the low fire performance requirements for materials used in road vehicles, with legislation "flaccid" compared to that applicable in railways, ships or aircraft.

"Post-collision fires in road vehicles, a pre-study" Ochoterena et al., SP Sweden report 2016:55 http://ri.diva-portal.org/smash/get/diva2:944435/FULLTEXT01.pdf and summary in International Fire Fighter Magazine, March 2017 http://iffmag.mdmpublishing.com/iff-magazine-issue-53-march-2017/

1.3 million fires per year in the USA

Nearly 1.3 million fires needing fire service intervention occurred in the US in 2014, according to latest data from the US Fire Administration (USFA). Fires caused over 3 400 deaths and nearly 16 000 injuries and cost nearly 12 billion US$. Losses resulting from fires were orders of magnitude higher than those from natural disasters such as floods or tornadoes. Three quarters of fires occurred in homes, and 16% of fire deaths occurred in vehicles.

"Fire in the United States 2005-2014", 18th Edition, January 2017, USFA and FEMA https://www.usfa.fema.gov/downloads/pdf/publications/fius18th.pdf and "Fire Risk in 2014", FEMA Topical Fire Report Series Volume 17, Issue 7/September 2016 https://www.usfa.fema.gov/downloads/pdf/statistics/v17i7.pdf

Low smoke non-halogen conduits for public safety

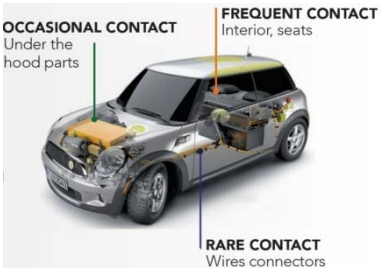
DELIKON, global specialist in conduit solutions for facilitating, housing and protecting cable installations in construction, continues to extend its range of "Low Smoke Halogen Free" (LSHF) products. These are particularly adapted to public buildings exposed to fire risks where protection of people, data and installations is paramount, such as transport terminals, shopping malls, banks, computer centres, schools, hospitals … The PIN flame retardant conduits ensure protection of cables in case of fire, so enabling electrical supply, communications and emergency systems (fire alarms, sprinkler controls, exit indications) to continue functioning. DELIKON emphasise that the non-halogen materials ensure lower emissions of smoke, acidity and toxic gases in case of fire. The DELIKON LSHF conduit range offers durability and long life-time reliability and includes heavy-duty steel reinforced conduits (crush proof), liquid tight conduits, smooth wipe-clean conduit exterior surfaces, light-weight and flexible ranges, conduits for automobile, chemical and oil industry applications, and a full range of liquid-tight connectors, corners, adapters and attachments.

DELIKON "Low Smoke Halogen Free Liquid Tight Conduit (LSHF-806) Zero Halogen", Ocotber 2016 http://www.delikon.com/lshf806.html

PIN self-extinguishing durable labels

CILS (Computer Imprintable Label Systems), international leading producer of labels for electronics, electrical components and other product, has launched CILS-8100FR durable fire-safe labels. The new labels are "durable" (resist chemicals and solvents, temperatures from -55 to +155 °C, abrasion, cleaning agents). CILS-8900FR (polyamide) labels resist up to nearly 400°C. The labels use PIN flame retardants to achieve fire safety standards of UL94 VTM-0 and VTM-01 and FAR smoke emission and toxicity requirements. The labels can be produced to any shape, size, format and design and can be printed using standard Thermal Transfer printing.

"CILS Launch 'Flame Retardant' self-extinguishing Durable Label Range", 16th March 2015
http://www.cils-international.com/usa/news/2015/03/16/cils-launch-%E2%80%98flame-retardant%E2%80%99-self-extinguishing-durable-label-range



ICL SAFR™ FR exposure and hazard assessment

ICL-TP has developed a methodology and tests to assess risks related to use of flame retardants in their applications (see also pinfa Newsletters n° 61 and 76), considering both their hazard and their exposure. The method takes into account four levels of substance hazard and three levels of exposure. Exposure is assessed based on frequency of exposure for a given use, and on potential emissions due to migration to the product surface (blooming), leaching or volatilisation. In all cases, the most likely emission and worst case exposure scenarios are used. The method presents the sustainability of different uses (identify uses that are recommended, acceptable or not recommended) or defines that the FR poses unacceptable hazards in which case alternatives must be identified. The company's objective is to enable users to select the most sustainable flame retardant solution for a given product or application.

SAFR® (Systematic Assessment for Flame Retardants) http://www.icl-group.com/sustainability/systematic-assessment-for-flame-retardants-safr/

Glass microspheres synergist for intumescent APP

Hollow glass microspheres (sodium borosilicate, 30-120 µm diameter) were tested as synergist with the intumescent PIN flame retardant, ammonium polyphosphate (APP) and pentaerithyritol (PER), in epoxy resin. 3 mm thickness epoxy resin sheets were tested with 20% total loading of flame retardant (of which 0 – 4% glass microspheres). Peak heat release of the epoxy sheets with flame retardants was around 1/3 of without, and was further reduced by around a quarter with 2% or 4% glass. UL94-V1 (3mm) was achieved with APP only, and V0 with 2% or 4% glass. Smoke production rate of epoxy with the PIN FR was less than half that of untreated epoxy, and the lowest smoke production rate was with 2% glass. The PIN FR also increased the epoxy's thermal decomposition temperature (TG thermogravimetric analysis), further increased with 2% glass. The authors conclude that glass microspheres act as an effective synergist to intumescent PIN flame retardant (APP), apparently by increasing density and compactness and reducing cracking of char.

"The synergistic effect of hollow glass microsphere in intumescent flame-retardant epoxy resin" J. Zhou et al., J Therm Anal Calorim, http://dx.doi.org/10.1007/s10973-017-6142-6

Glass microspheres synergist in polyurethane

Hollow glass microspheres HGM (sodium borosilicate, 15-150 µm diameter) were tested for flame retardancy in thermoplastic polyurethane (TPU), supplied by Bayer. Results showed that 2% loading of HGM glass reduced total spoke release by 26%, increased LOI by around 10%, reduced peak heat release rate (PHRR) by up to 70% and enabled achievement of UL94-V1 (vs. V2 for pure TPU). The authors conclude that the glass microspheres act by catalysing TPU carbonisation, and so char formation, and by modifying the char structure.

"Fire hazard reduction of hollow glass microspheres in thermoplastic polyurethane composites", Journal of Hazardous Materials 2017, C. Jiao et al., http://dx.doi.org/doi:10.1016/j.jhazmat.2017.02.019

High reliability disc capacitors

Vishay Intertechnology is a global leader in electronic components (semiconductors, resistors, capacitors …), since 1962. The company's new compact ceramic disc capacitors are designed to ensure extremely high reliability and are the first in industry to offer pulse strength of 10kV, resist temperatures to 85°C and relative humidity of 85% (at rated voltage for 1 000 hours). Compact design enables diameters down to 7.5 mm for use in power supplies, smart meters, white goods, industrial equipment and consumer electronics. These levels of performance are achieved including with a Vishay Green encapsulation resin with UL94-V0 fire performance using PIN flame retardants, conform to JEDEC® JS709B "low halogen" standard.

www.vishay.com and "Vishay – Compact series ceramic disc capacitors offer extremely high reliability" 8 February 2017
http://www.vishay.com/company/press/releases/2017/170206VY1CompactSeries/
JEDEC 709B 'Definition of "Low-Halogen" For Electronic Products' as a Public Available Specification' see pinfa Newsletter n° 66 and https://www.jedec.org/standards-documents/results/709B

Zirconium N P flame retardant for polyamide

An inorganic (zirconium) - phosphorus (P) - nitrogen (N) PIN flame retardant was experimentally developed and tested in polyamide (PA6). The FR was produced in a one-step reaction from α-ZrP (alpha zirconium phosphate) at 3 – 30%, melamine and cyanuric acid. The α-ZrP-melamine cyanurate (α-ZrP-MCA) was then blended with PA6 using a screw extruder. UL94-V0 was achieved at 12% loading of FR (with α-ZrP > 5%) and fire performance was significantly better than PA6 without FR or PA6 with melamine cyanurate only (no α-ZrP). SEM (scanning electron microscopy) showed that the integration of α-ZrP results in a more compact, homogenous, ceramic-like char, acting as an effective barrier against fire. The α-ZrP-MCA PIN flame retardant showed not only to improve the fire performance of polyamide, but also to improve crystallinity and to maintain good mechanical properties.

"Effects of α-ZrP on Crystallinity and Flame-Retardant Behaviors of PA6/MCA Composites", Y. Xiao, Int J Polymer Science, 2017, Article ID 6034741 https://doi.org/10.1155/2017/6034741

Fish waste DNA and clay PIN FR for epoxy

DNA (deoxyribonucleic acid) from fish waste (herrings) was combined in a one-step reaction with commercially available modified silicate (montmorillonite) nanoclay and tested as a PIN flame retardant in epoxy resin. Around 0.7% w/w DNA was integrated into the clay using clay as slurry and acetone solvent at pH2, expanding the *d*-space between clay layers by c. 50%. The integration of DNA into the clay improves dispersion into and interactions with the epoxy resin, and contributes to flame retardancy because DNA is an organo-carbon phosphorus-based chemical. 2.5% loading of the DNA-clay product improved tensile strength, tensile modulus and toughness of the epoxy and reduced peak heat release rate (PHRR) by around 25%, in all cases compared to both epoxy without clay and to clay only, no DNA.

"Fish DNA-modified clays: Towards highly flame retardant polymer nanocomposite with improved interfacial and mechanical performance", O. Zabihi et al., Nature Sci Reports, 6:38194, DOI: 10.1038/srep38194, 2017
https://polymerandfire.files.wordpress.com/2016/12/srep38194.pdf

Biological gene bases as PIN FR synergists

Four different nitrogen containing bases (adenine, guanine, cytosine and uracil) were tested as PIN flame retardant combinations with APP (ammonium polyphosphate) in polypropylene. APP was supplied by Presafer and the bases by Aladdin. These bases are the same molecules as naturally found in all living cells in genetic material (DNA, RNA). Polypropylene containing 1-2% cytosine or uracil and 16-17% APP achieved UL94-V0, with no drip, and LOI of nearly 28% or higher (that is, fire performance comparable to 25% APP loading). Adenine and guanine were not so effective. The flame retardancy effect of the bases cytosine and uracil is identified to be due to char generation, with improved thermal stability of the char at high temperatures. Uracil also reduced the peak heat release rate and total heat release (PHRR, THR).

"Regulating effects of nitrogenous bases on char structure and flame retardancy of polypropylene/intumescent flame retardant composites", ACS Sustainable Chemistry & Engineering, 2017, Z. Wang et al., http://dx.doi.org/10.1021/acssuschemeng.6b02712

Heat-resistant DOPO derivative for engineering plastics

Three molecules of DOPO were reacted together, in a two-stage process, to give a star-shaped DOPO derivative (GL-3DOPO) containing just over 10% by weight phosphorus. This product showed resistance to thermal composition up to 360°C (considerably higher than DOPO, which begins to decompose at around 200°C), so enabling compatibility with processing temperatures of engineering polymers. The GL-3DOPO showed good blending compatibility and to be a plasticiser in polar polymers. 25% GL-3DOPO loading enabled achievement of UL94-V0 in PET, PBT, polycarbonate, and polyamines 6 and 66. 8% only GL-3DOPO enabled UL94-V0 in PET, with formation of a compact char layer, suggesting that the molecule acts in both the solid and the gas phase.

"Synthesis of a heat-resistant DOPO derivative and its application as flame-retardant in engineering plastics", M. Xie et al., J Applied Polymer Science 2017
http://dx.doi.org/10.1002/app.44892

Nanoclay PIN FR for polyester construction panels

Prefabricated polymer panels can provide low-cost, light-weight, structural elements for rapid construction of housing or modular office buildings, but pose fire safety dangers if not flame retarded. Infusion moulded, glass-fibre reinforced polyester (GPRP) panels, 3 or 4 mm thickness, were produced from polyester resin, reinforced with six layers of 410 g/m^3 biaxial E-glass, pre-treated to be compatible with polyester resin. Montmorillonite nanoclay at 5% loading was tested as fire safety treatment. The clay had average particle size c. 10 µm and was surface treated with dimethyl dialkyl amine to enable resin compatibility. The GRFP panels were tested for fire performance according to ISO 9750-1:2013, as required by the Australian building code, that is 4 mm GFRP panels were sandwiched either side of 12mm of polyisocyanurate foam, and also in a simulation of installation in a Netherlands office building. The nanoclay resulted in considerable reductions in heat release rates and proved sufficient to prevent fire developing to flash over in room tests.

"Fire performance of prefabricated modular units using organoclay/glass fibre reinforced polymer composite", Q. Nguyen et al., Construction and Building Materials 129 (2016) 204–215 http://dx.doi.org/10.1016/j.conbuildmat.2016.10.100

Other News

Ellen MacArthur Foundation pushes for systemic plastics recycling: the New Plastics Economy, led by the Foundation with partners from the polymers and chemicals industry, supermarkets, the food industry and the waste sector, proposes a new approach to transform global plastics flows towards a circular economy. Losses of plastics which are not recycled are estimated at around 100 billion USD/year globally, and environmental impacts of these losses (externalities) at 40 billion. The project centres on packaging plastics, as the biggest potential, but also underlines the need to avoid substances with environment or health concerns which can then pose issues in recycled plastics. Foundation proposes a global plastics protocol, to redesign products, packaging and systems to facilitate collection and recycling and also reuse, for example avoiding multi-material packaging.

Ellen MacArthur Foundation, New Plastics Economy https://newplasticseconomy.org/

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For abbreviations see: www.pinfa.org