

EXPANSION OF POLYOLEFIN APPLICATIONS VIA INTUMESCENT HALOGEN-FREE FLAME RETARDANT TECHNOLOGY



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Polymer Additives R&D Laboratory
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Introduction

2

Flame Retardancy of Intumescent Flame Retardant

3

Combining Flame Retardancy with Thermal Stability

4

Summary

Outline



| | |
|----------------|-----------------------------------|
| Establishment | : 1917 (as Asahi Denka Kogyo) |
| Business field | : Chemicals, Foods, Life sciences |
| Capital | : 22.9 billion¥ |
| Affiliates | : 40 (Japan 18, Foreign 22) |
| Employees | : 5,154 (group total, 2019.3) |
| TB of FY2018 | : 299.3 billion¥ |



ADEKA HQ
(Front : Ogu R&D site)

Core Business : Polymer Additives, Foods



Functional Chemicals
Polymer Additives
Lubricants etc.



**IT and Electronic
chemicals**
Semiconductor
Circuit materials etc.



Commodity Chemicals
Polyalcohol etc.



Foods
Bread and oil ingredients etc.

■ Additives for polyolefins and Engineering plastics

- Antioxidants
- Light stabilizers
- Nucleating agents
- Clarifiers
- Metal deactivators
- Flame retardants



Flame retardants



Light stabilizers/
Nucleating agents



Clarifiers

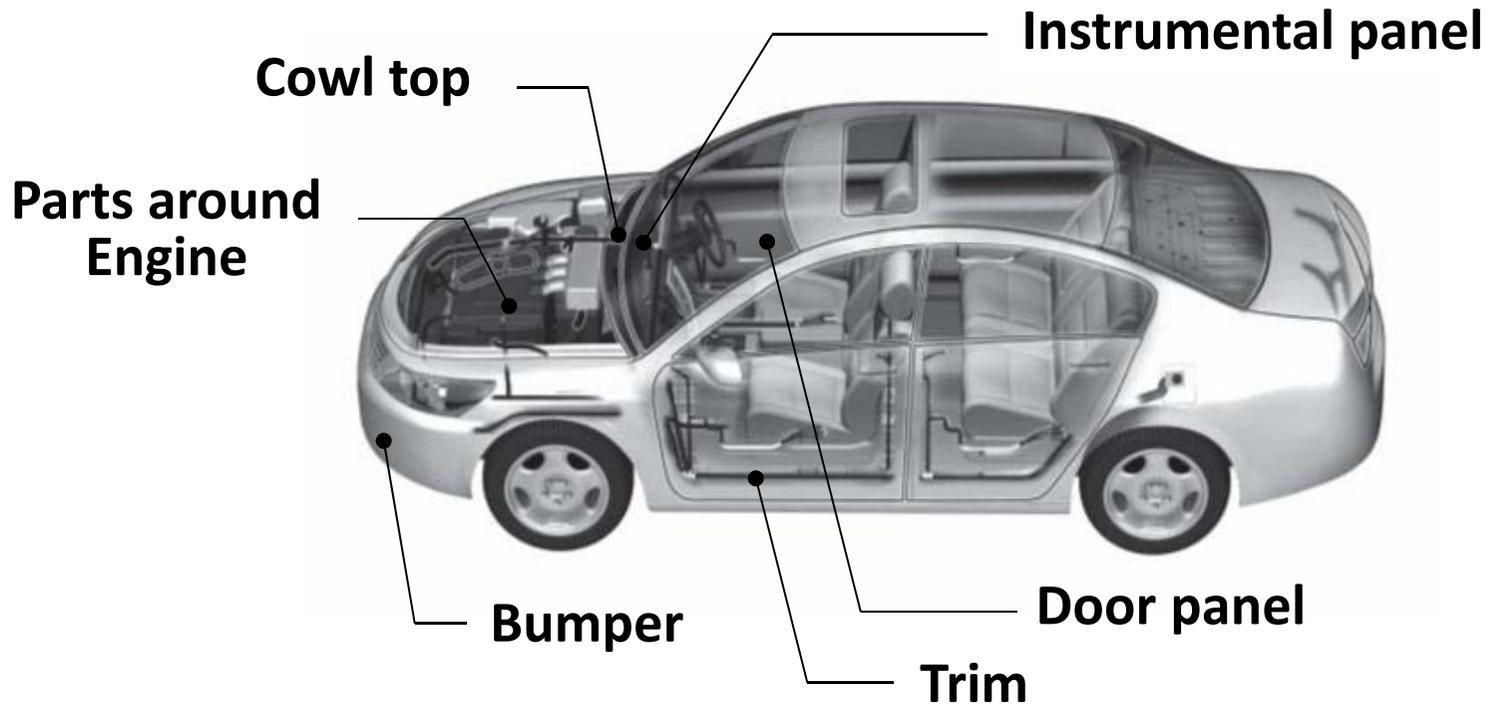
■ PVC stabilizers & plasticizers

- Stabilizers
- Plasticizers



Lead & Tin free PVC stabilizers

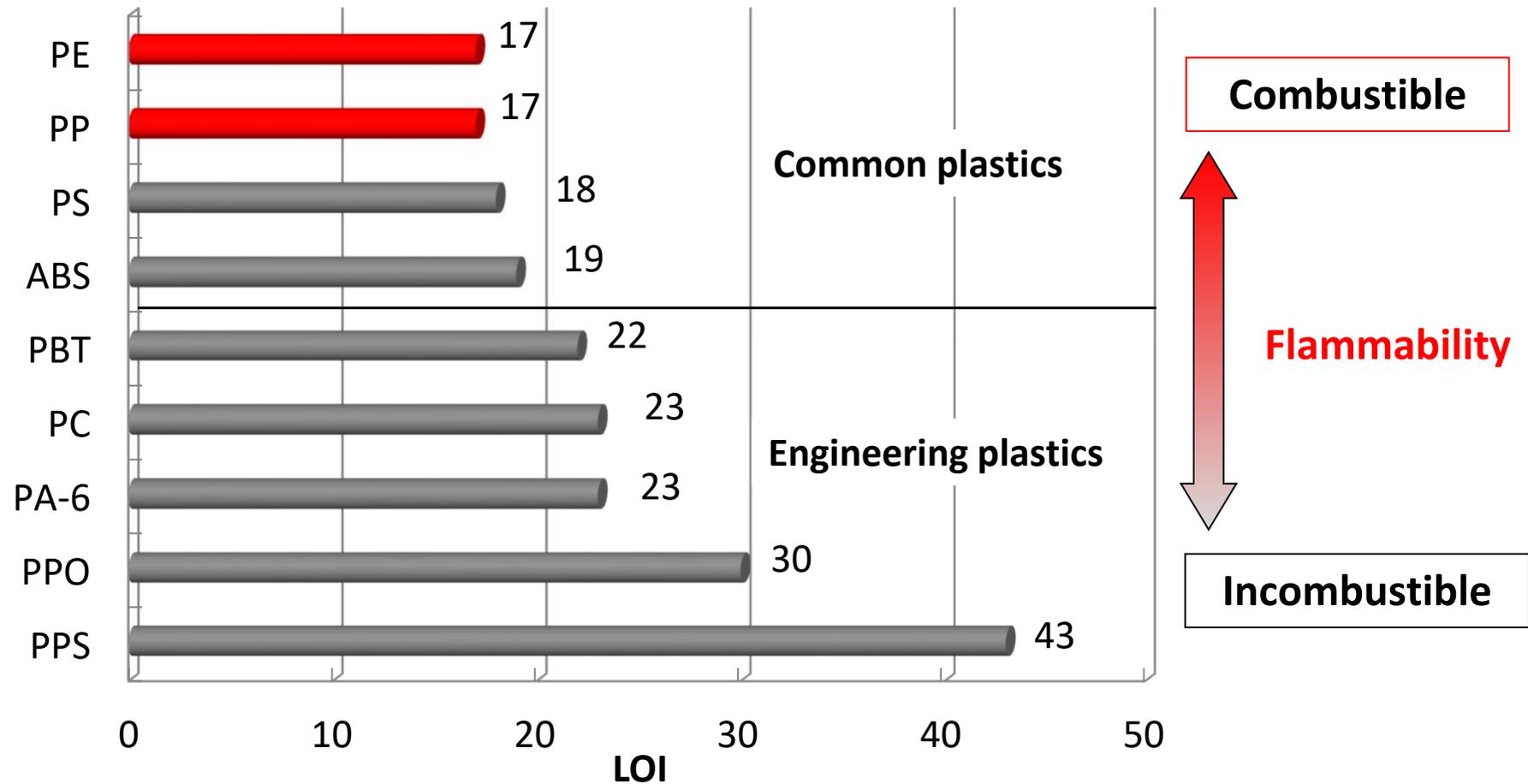
Typical Automotive Parts made from Polyolefins



Superiority of Polyolefins

- Low density
- Superior mechanical properties
- Excellent chemical resistance
- Recyclability
- Ease of processing and molding
- Low cost, etc.

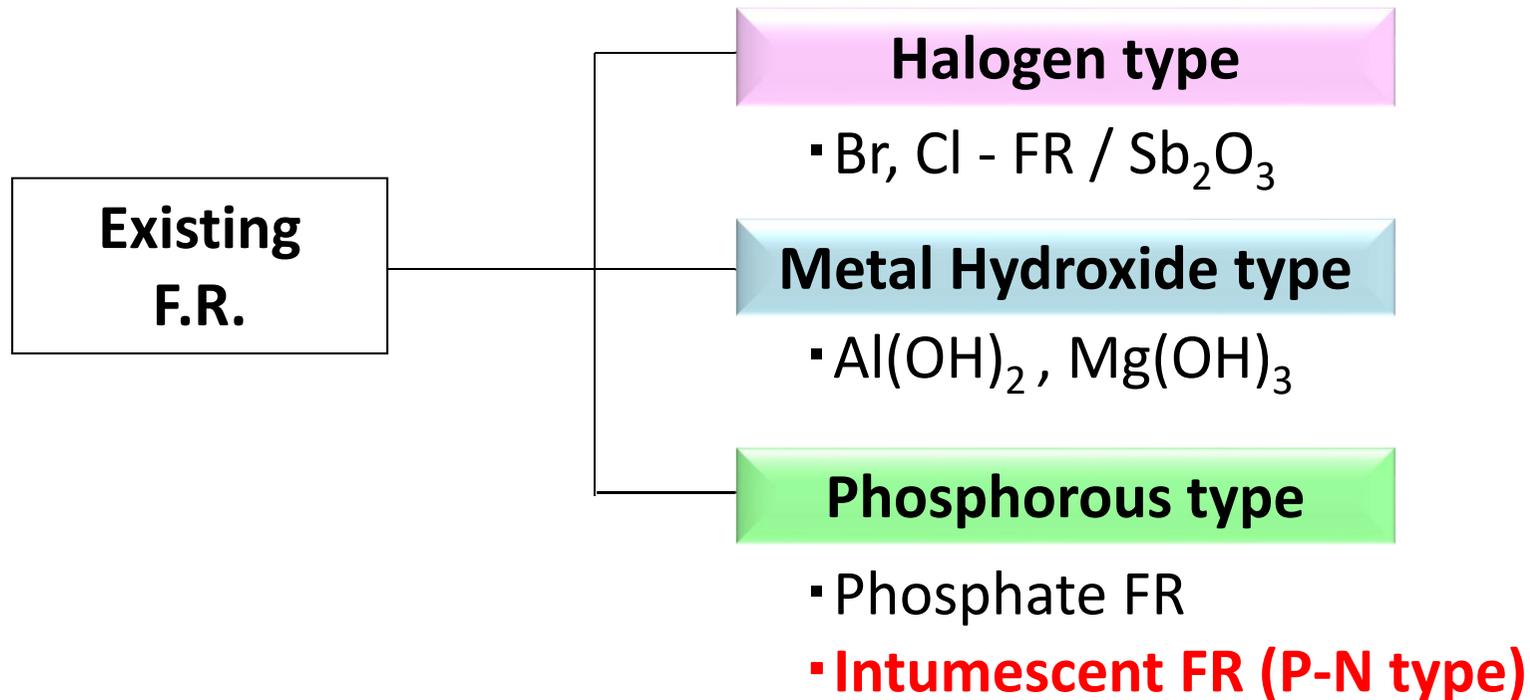
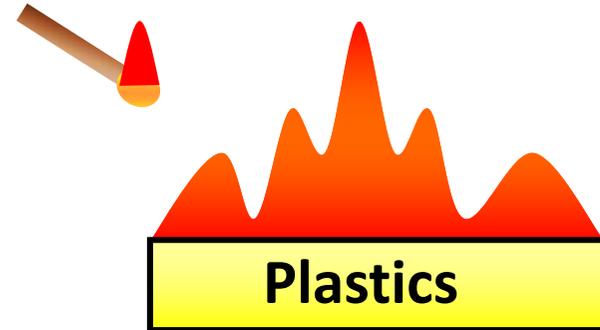
Limiting Oxygen Index(LOI) of Polymers



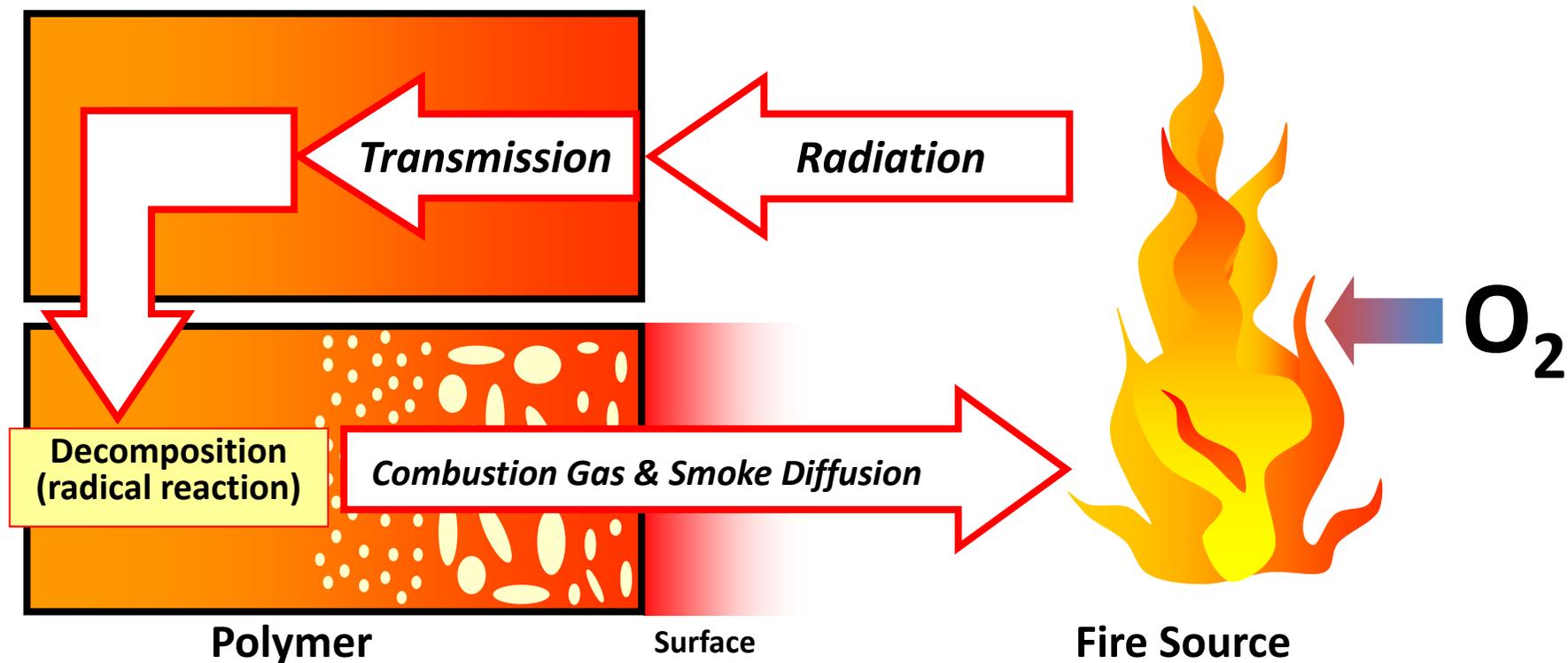
***Polyolefins are very combustible
Flame retardants are necessary for polyolefins***

What is Flame retardant?

*Additive to Inhibit
the combustion of plastics*



Mechanisms of Polymer Combustion



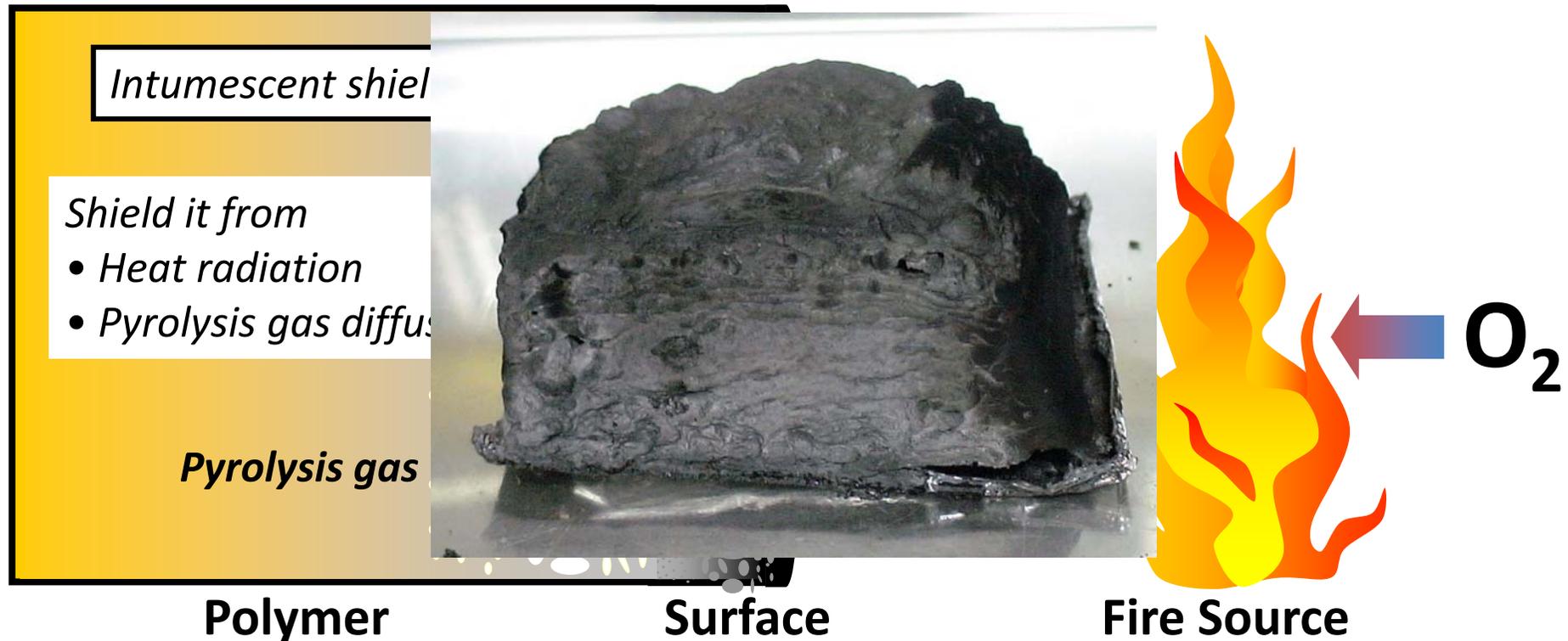
To provide flame retardancy to polymer



Inhibit or even suppress the combustion process

Intumescent FR (Halogen Free)

- Intumescent system is a shield formation on the polymer surface



Intumescent system blocks radiation and depresses pyrolysis gas diffusion.

Features of Intumescent Flame Retardant

- Low smoke
- Low toxic gases generation (CO, HBr etc.)

Combustion Behavior of Flame Retarded PP

Both samples are classified UL-94 V-0, but burning behavior differs :



Br-FR / Sb_2O_3

- ✓ Strong Fire
- ✓ Dark smoke



Intumescent(Int.)-FR*

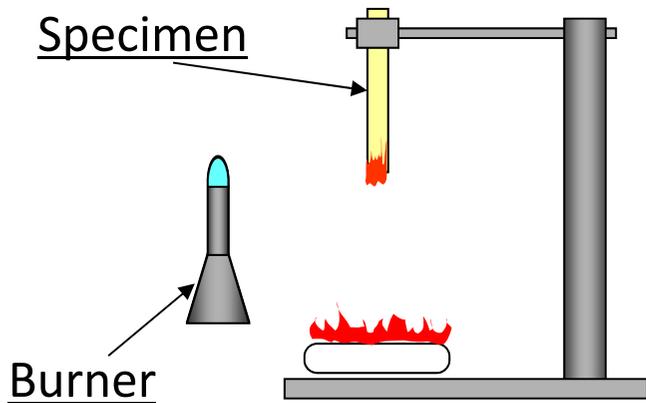
- ✓ Weak Fire
- ✓ Intumescent Layer

These combustion behaviors are notably different with respect to the fire intensity and smoke.

- 1 Introduction of Intumescent Flame Retardant
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- 3 Combining Flame Retardancy with Thermal Stability
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Dosage to achieve V-0 rating

UL-94V test



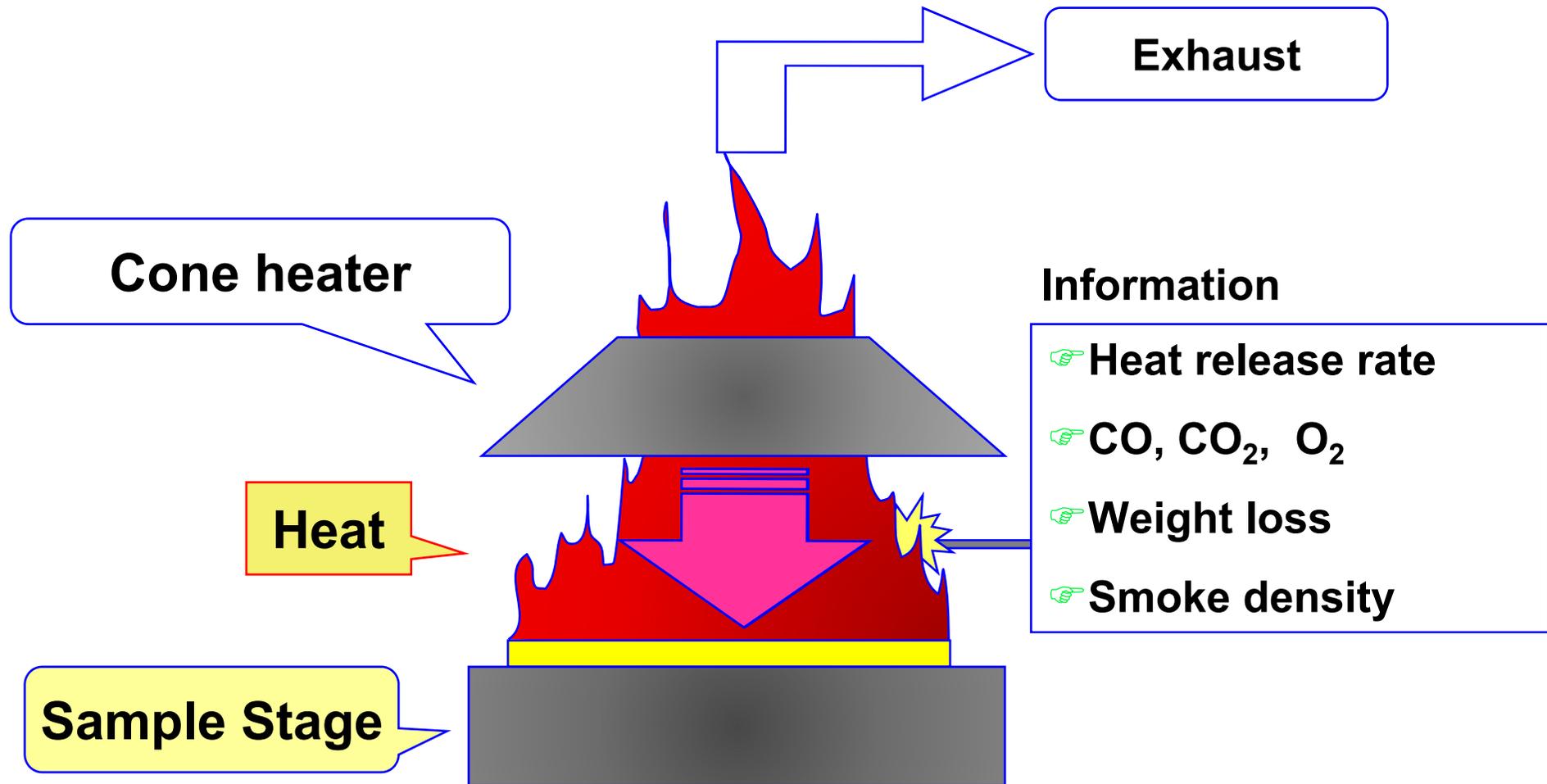
| | |
|-----------------|------------------|
| Ignition source | 20 ± 1 mm |
| Specimen | Bar |
| Ignition | 10 (s) × 2 times |

Ranking
 (Good) V-0 > V-1 > V-2 > N.R. (Bad)

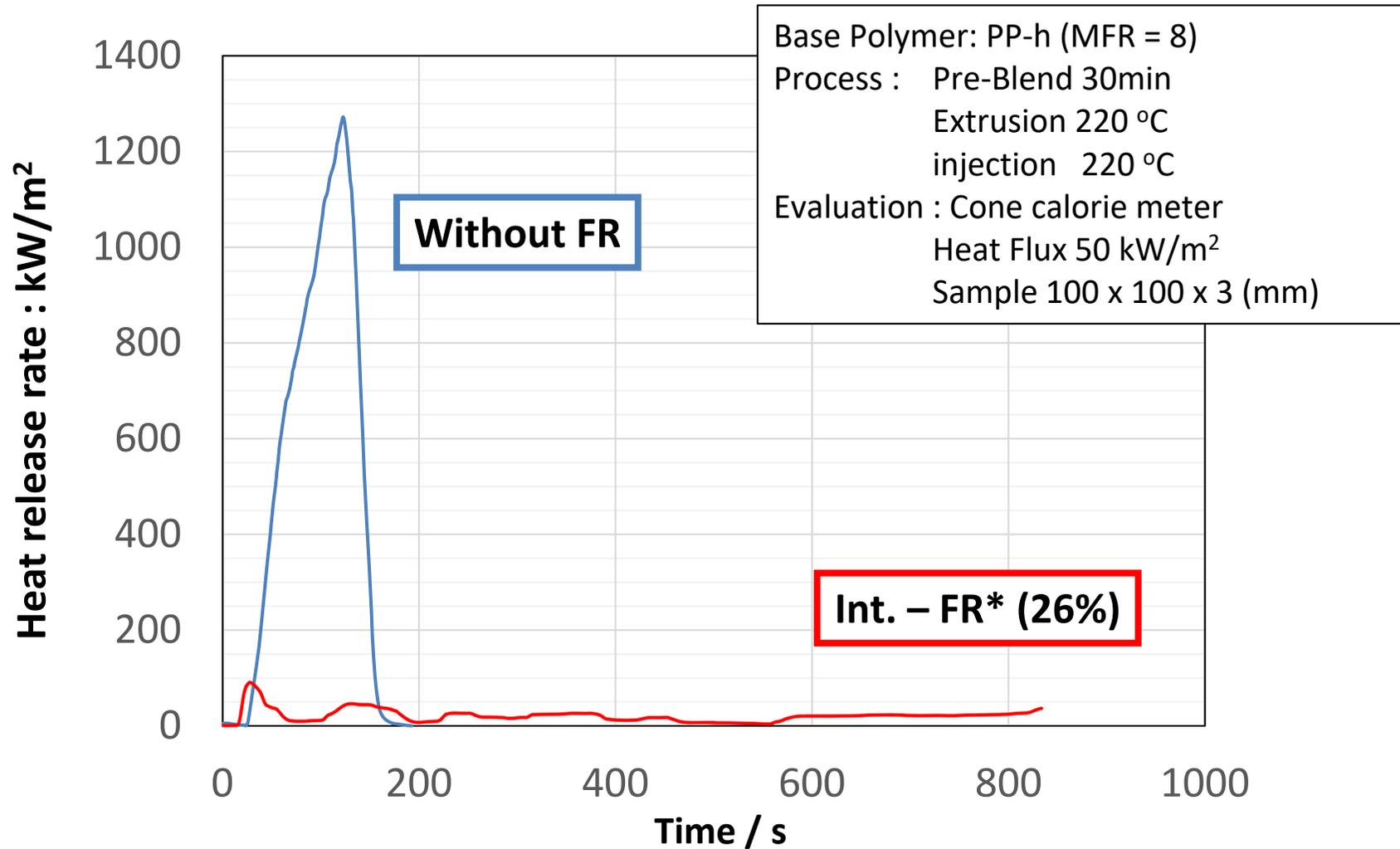
Resin : ICP
 Additives : AO-60 / 2112 / CaSt = 0.1 / 0.1 / 0.1 phr

| | @1.6mm | ICP | | | |
|----------------------|--------|------|------|------|------|
| | | MFR | 1.6 | 8 | 14 |
| Dosage of Int.-type* | 32 % | | | V-0 | V-0 |
| | 30 % | | | V-0 | V-2 |
| | 28 % | | | V-0 | V-2 |
| | 26 % | | V-0 | V-2 | V-2 |
| | 24 % | V-0 | V-2 | V-2 | V-2 |
| | 22 % | V-0 | V-2 | V-2 | V-2 |
| | 20 % | N.R. | V-2 | V-2 | V-2 |
| | 18 % | N.R. | N.R. | N.R. | N.R. |

Cone calorimeter measurement (ISO 5660-1)



Heat Release Rate



Intumescent FR could depress HRR, dramatically.

Smoke Opacity and Smoke Toxicity (ISO 5659-2)

The assessment of smoke opacity and smoke toxicity has been carried out according to ISO 5659-2 (Smoke generation test) standard. The materials have been tested according to two degradation modes:

- 1) Irradiance of 25 kW/m^2 with a pilot flame
- 2) Irradiance of 50 kW/m^2 without pilot flame

These fire tests methods are part of the European Railway standard EN 45545-2.



Figure : Pictures of the cone heater (left) and the single smoke chamber (right) for ISO 5659-2 test
(Source: External institute)

Smoke Opacity & Toxicity (25 kW/m²)

Heat Flux: 25 kW/m² with pilot flame
 Thickness: 3mm
 Base Polymer: PP-b
 Additives: GMS/AO-60/2112/Ca stearate = 0.3/0.1/0.1/0.1

| Formulation | Ds** Max | Smoke Toxicity and detected gases at 8 min | | | | |
|----------------------|-------------|--|---|----------------------------|-----------------------------|---|
| | | CIT _G (Toxicity Index) | CO ₂ (mg/m ³) | CO (mg/m ³) | HBr (mg/m ³) | SO ₂ (mg/m ³) |
| Control (PP only) | 602 | 0.10 | 33,402 | 645 | ND*** | ND |
| Intumescent Type* | 100 | 0.01 | 4,450 | 79 | ND | ND |

* The dosage which achieves V-0 classification

** Ds max: maximum specific optical density *** ND: Not detected

Intumescent FR could depress not only smoke generation but provides also low CIT_G. Toxic gas generation is low as well.

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Intumescent Flame Retardant

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with Thermal Stability

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Further Investigation on Thermal Stability

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Summary

Applications for which Thermal Stability is Required

Automotive



Materials used in battery periphery
Materials used for control panel

For these applications, both flame retardancy and long thermal stability should be required.

Intumescent FR gives excellent thermal stability and shows synergistic effects with specific antioxidants.

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Formulation

Formulation

resin Impact copolymer, MFR = 14

FR Int. – FR(ADK STAB FP-2500S) : 28 % for V-0 on UL94@1.6 mm

Additives ADK STAB 2112 ¹⁾ / ADK STAB AO-60 or AO-20 ²⁾ / Ca-St / GMS
= 0.1 / 0.1 / 0.1 / 0.3 %

1) Phosphite type Anti-Oxidant

2) Phenol type Anti-Oxidant

Process conditions

Extrusion : Twin screw extruder at 220 °C

Injection : at 220 °C (Mold : 40 °C)

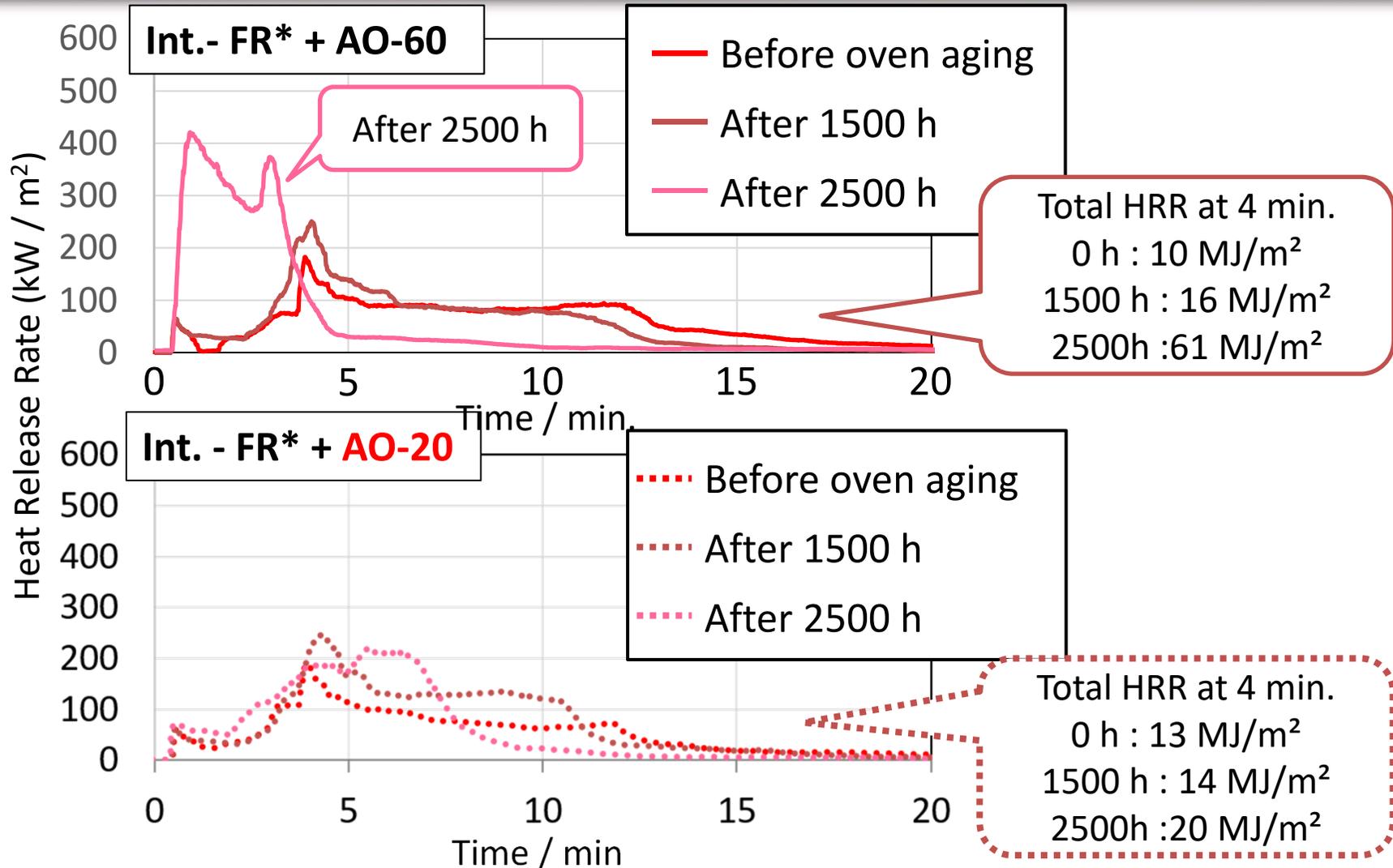
Oven aging

at 150°C (circulation air oven)

Evaluation

Cone Calorie Meter at 50 kW/m²

Flame Retardancy after Oven Aging at 150 °C



The Combination of Intmescent FR with ADK STAB AO-20 retained its flame retardancy longer.

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Intumescent flame retardant provides excellent performance :

1. Flame retardancy

*Reducing the overall heat release rate
and smoke opacity and smoke toxicity*

2. Expand applications by additive combination

Excellent thermal stability in combination with advanced AO

ADEKA's Product range of Intumescent Flame Retardants

| Name | Feature |
|--------------------|--------------------------|
| ADK STAB FP-2100JC | Higher Thermal Stability |
| ADK STAB FP-2500S | Higher Flame Retardancy |



Thank you for your attention