



Development and characterization of bio-based flame retardant materials

May 25th 2011, Ghislenghien

*Unité Matériaux et Transformations (UMET) - CNRS UMR 8207
Equipe Ingénierie des Systèmes Polymères
Ecole Nationale Supérieure de Chimie de Lille (ENSCL) - France*

Outline

Intumescent process

**Bio-based
char formers**

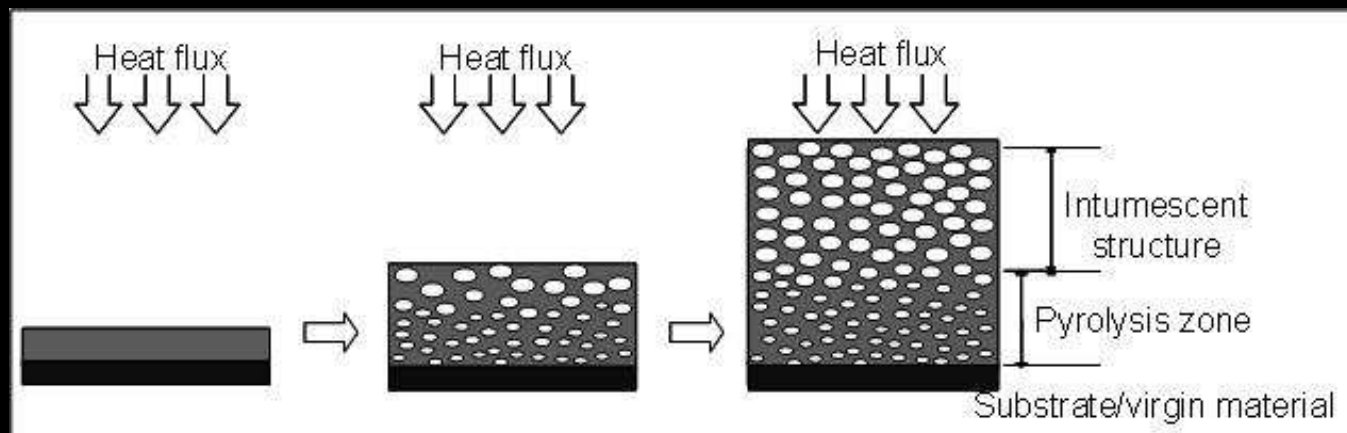


**Flame
retardancy
of PLA?**

**Bio-based
acid sources**

Developments

Intumescence



COMPOSITION :

acid source / carbon source / blowing agent

ammonium polyphosphate
(APP)

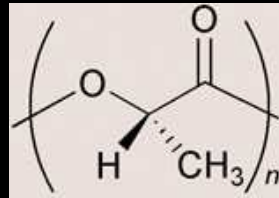
pentaerythritol
(PER)

ammonium polyphosphate
(APP)

use of bio-based additives



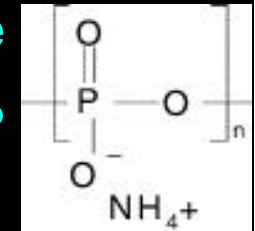
Preparation of materials



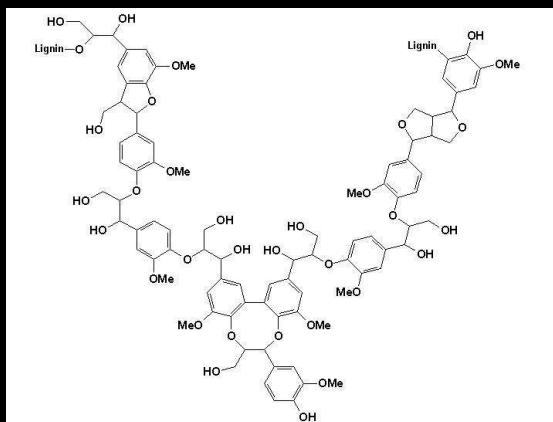
Polymeric matrix
PLA – 60%
(Cargill)



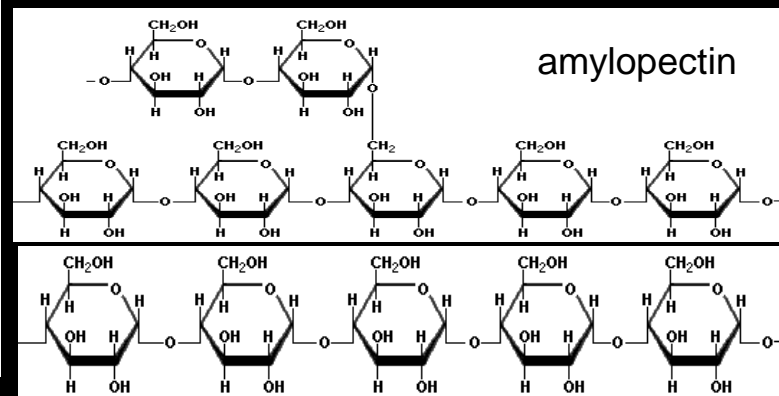
Blowing agent + acid source
APP – 30%
(Clariant)



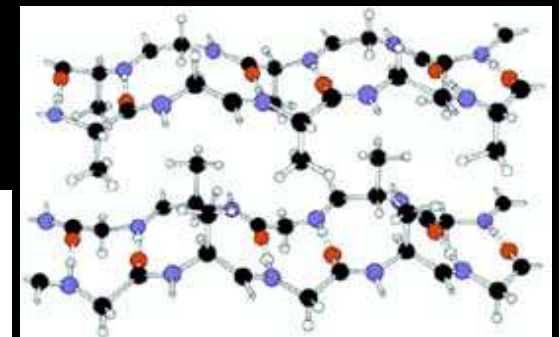
Char formers – 10%



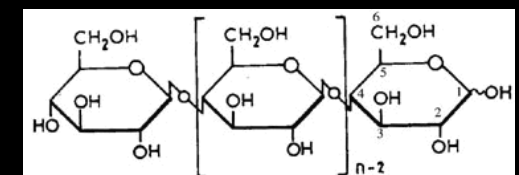
Lignin



Starch



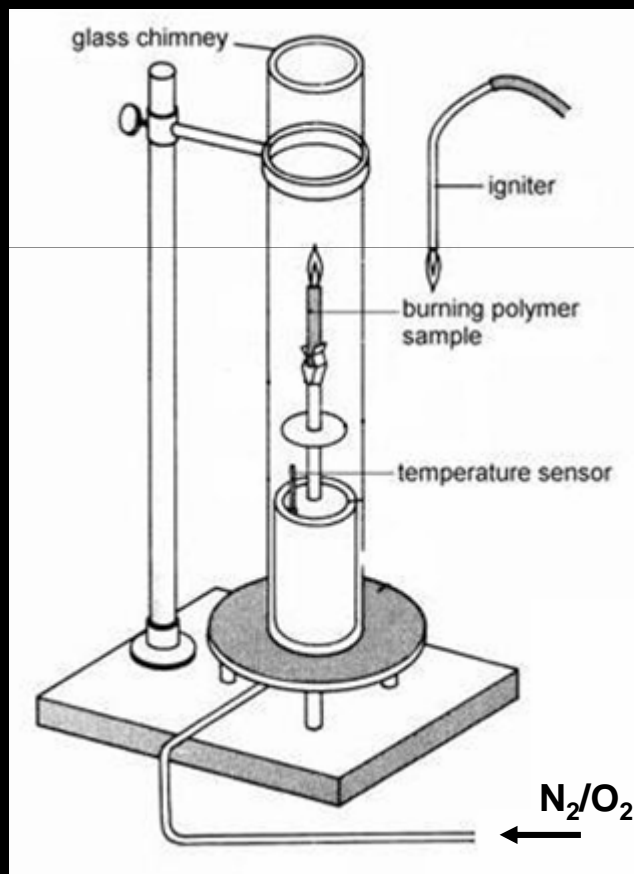
Keratin



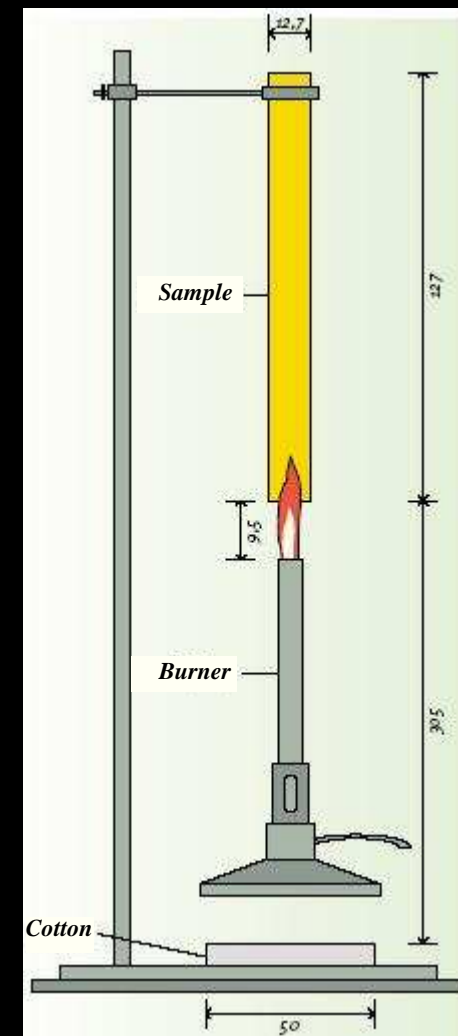
Cellulose

Fire Tests

LOI: Limiting Oxygen Index

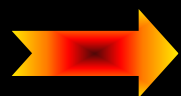


UL-94 test



Fire Retardant Properties

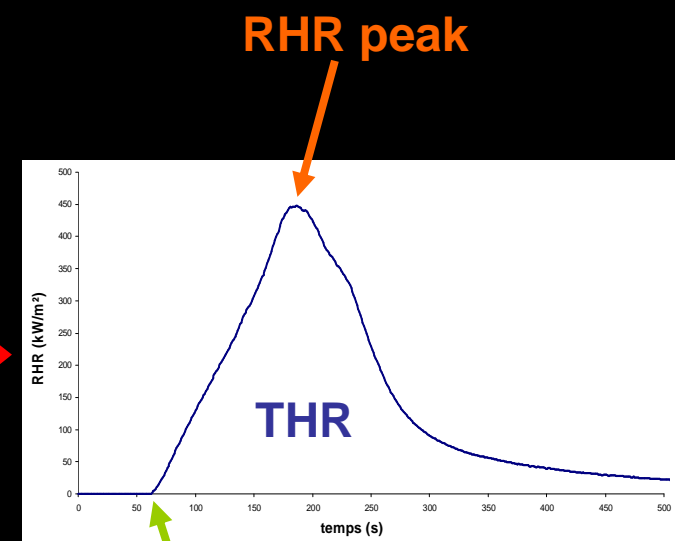
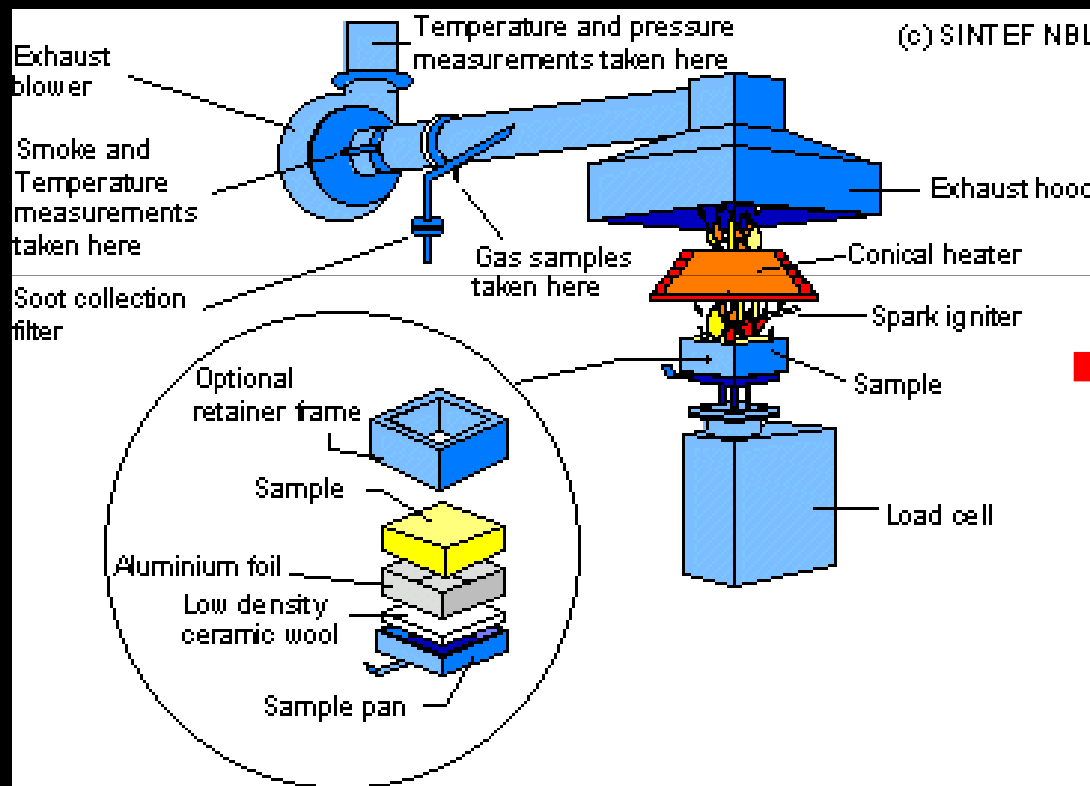
Formulations	LOI	UL-94
100 % PLA	21	non classified
70%PLA + 30%APP	37	V2
60%PLA + 30%APP + 10%PER	58	V2
60%PLA + 30%APP + 10%lignin	32	V0
60%PLA + 30%APP + 10%starch (maize)	47	V0
60%PLA + 30%APP + 10%starch (potato)	44	V0
60%PLA + 30%APP + 10%cellulose	41	V2
60%PLA + 30%APP + 10%keratin	44	V2



Most efficient carbon sources: lignin and starch

Fire test

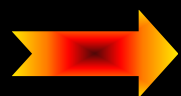
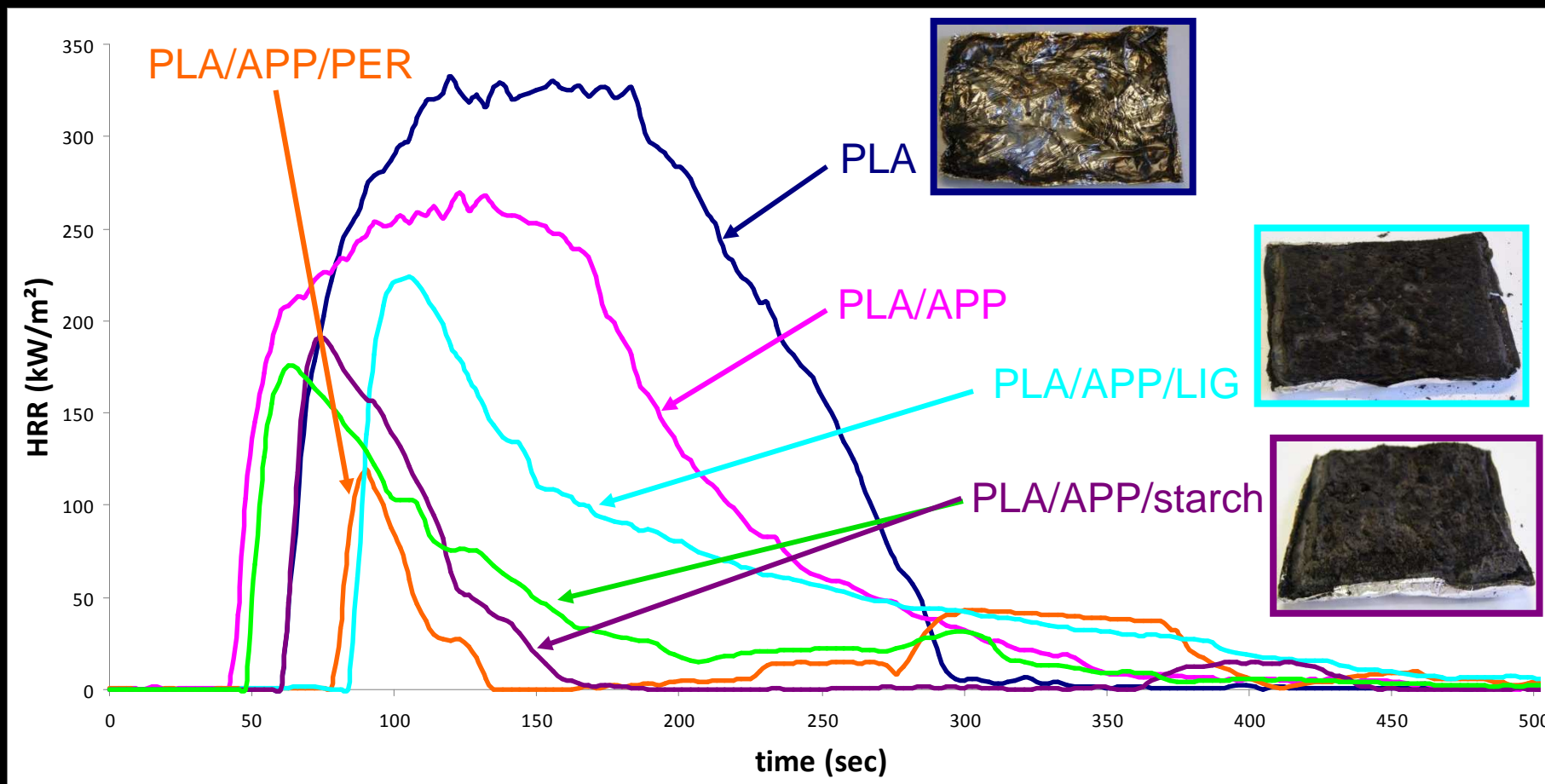
Cone calorimetry (ASTM D 3801 or ISO 5660)



Time of ignition

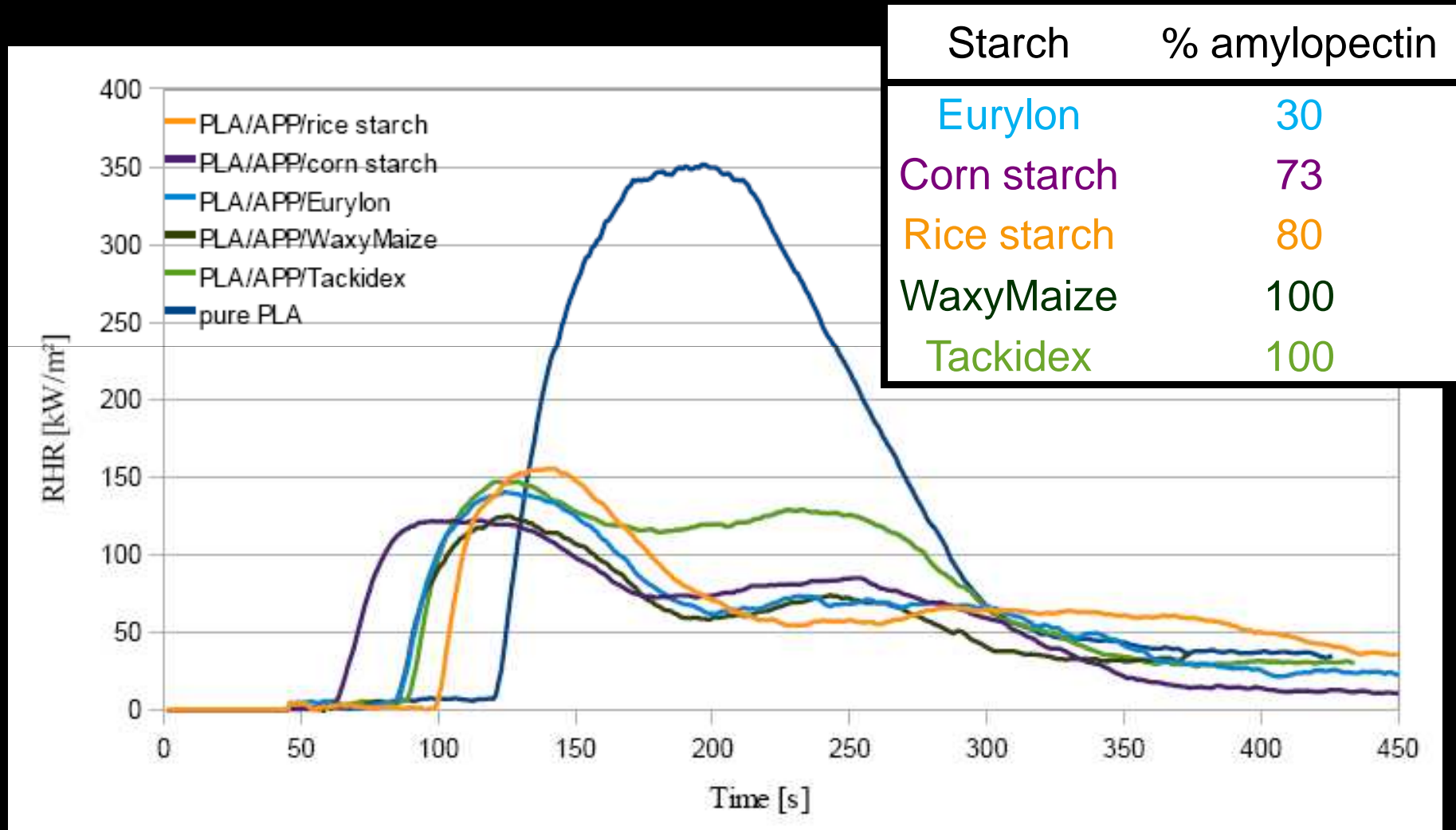
Heat flux = 35 kW/m²

Fire Retardant Properties



Decrease of the HRR with lignin and starch

Influence of starch composition



Fire Retardant Properties

Formulations	pRHR (kW/m ²)	∇ pRHR (%)	THR (MJ/m ²)	LOI
PLA	350		48	21
PLA/APP/Eurylon	140	60	24	44
PLA/APP/corn starch	122	65	24	47
PLA/APP/rice starch	155	56	26	48
PLA/APP/waxy starch	125	64	19	44
PLA/APP/Tackidex	147	58	29	42

Dispersion of additives

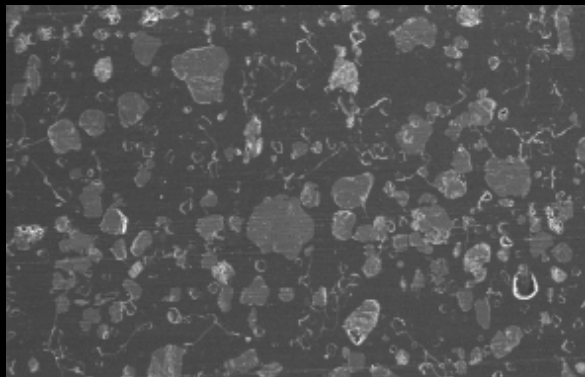
Rice starch



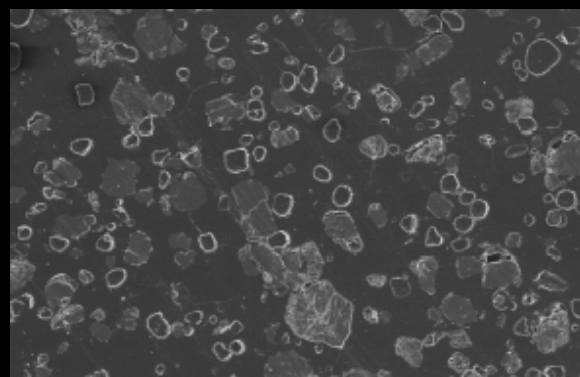
Eurylon



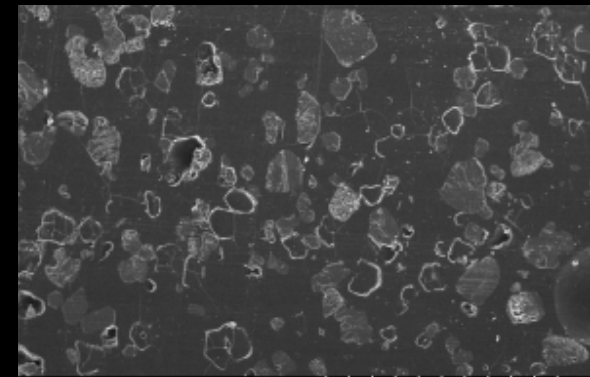
Corn starch



GaN-Qdots 6.0kV 12.0mm x450 SE(M) 100um

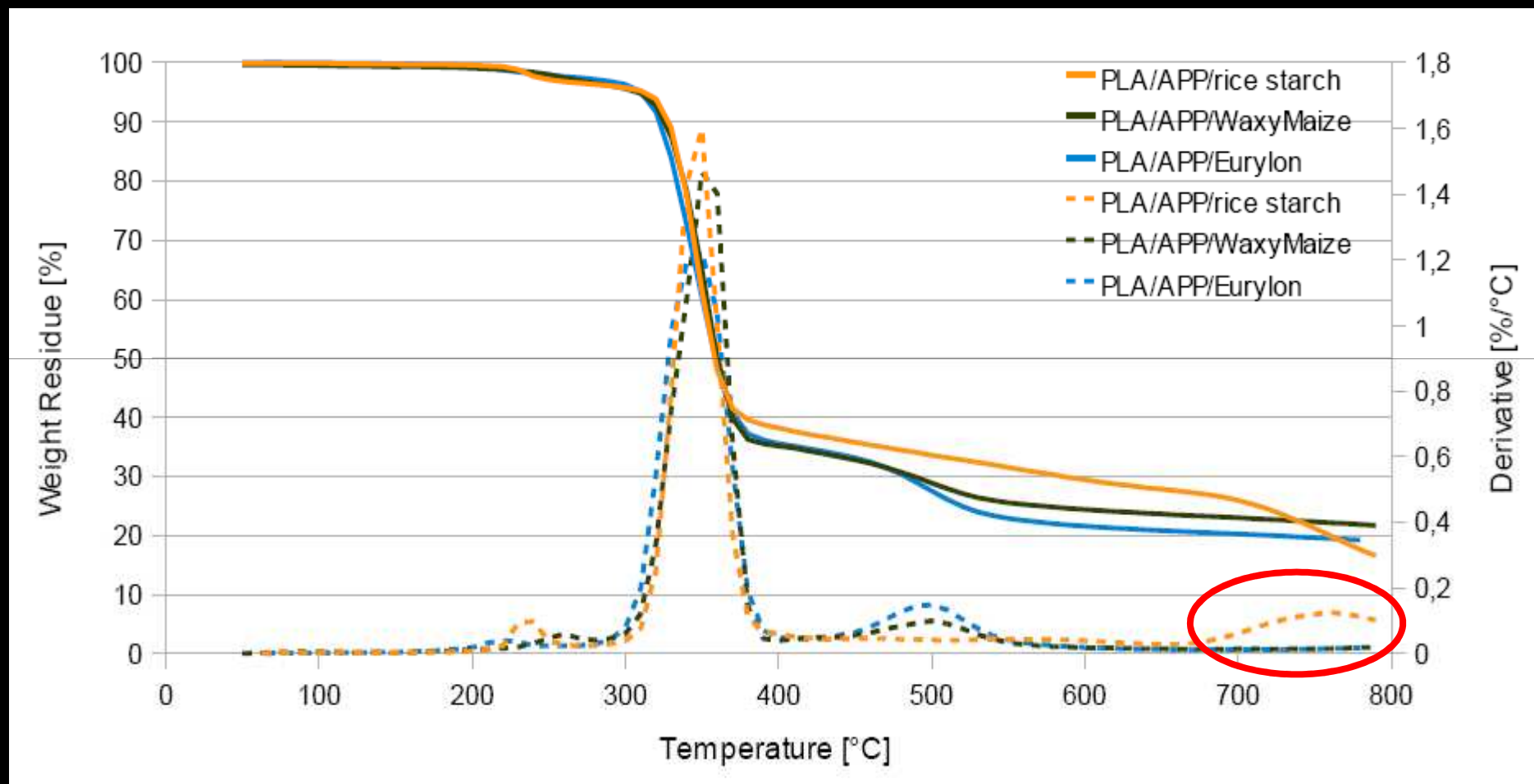


GaN-Qdots 6.0kV 12.1mm x450 SE(M) 100um



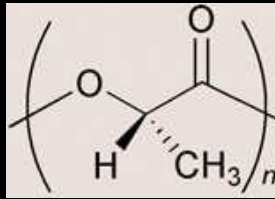
GaN-Qdots 6.0kV 12.1mm x500 SE(M) 100um

Thermal stability



Better stability of the PLA/APP/rice starch system at high temperatures

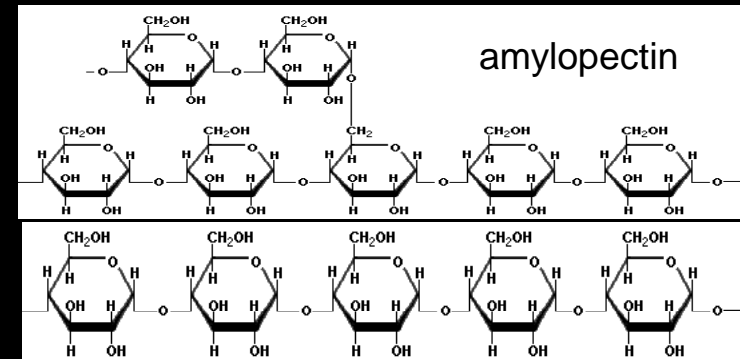
Preparation of materials



Polymeric matrix
PLA – 60%
(Cargill)



Char former – 28%

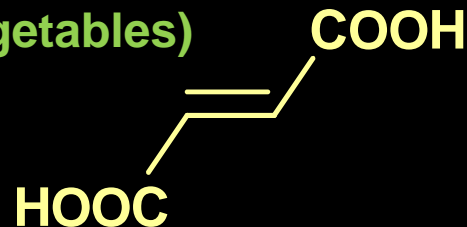


Starch

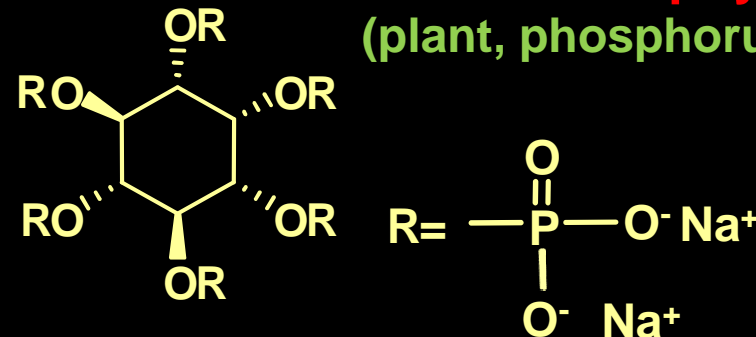
amylose

Blowing agent + acid source
APP + bio-based acid source
6% + 6%

Fumaric acid
(fruit and vegetables)

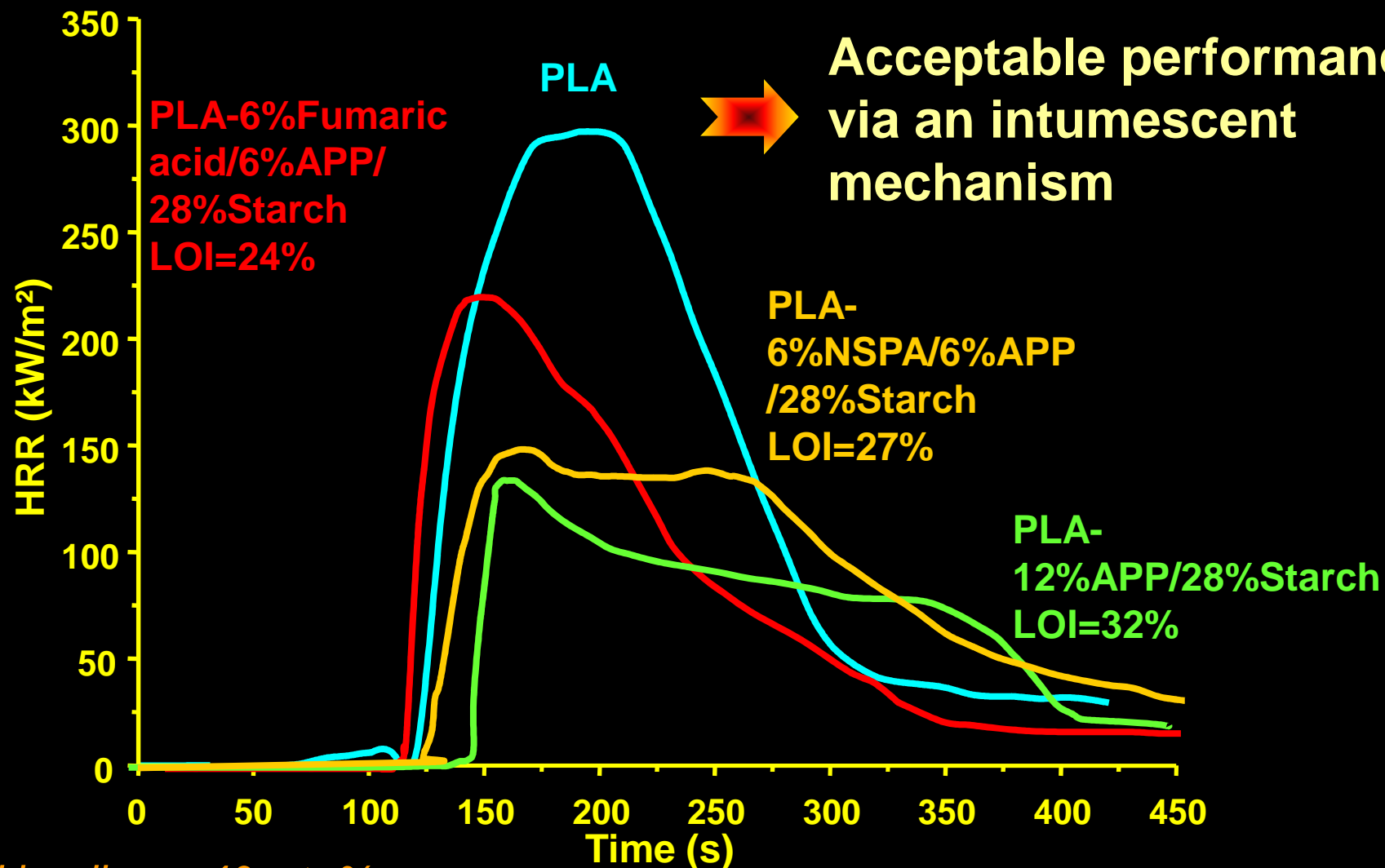


Na salt of phytic acid
(plant, phosphorus storage)



Fire Retardant Properties

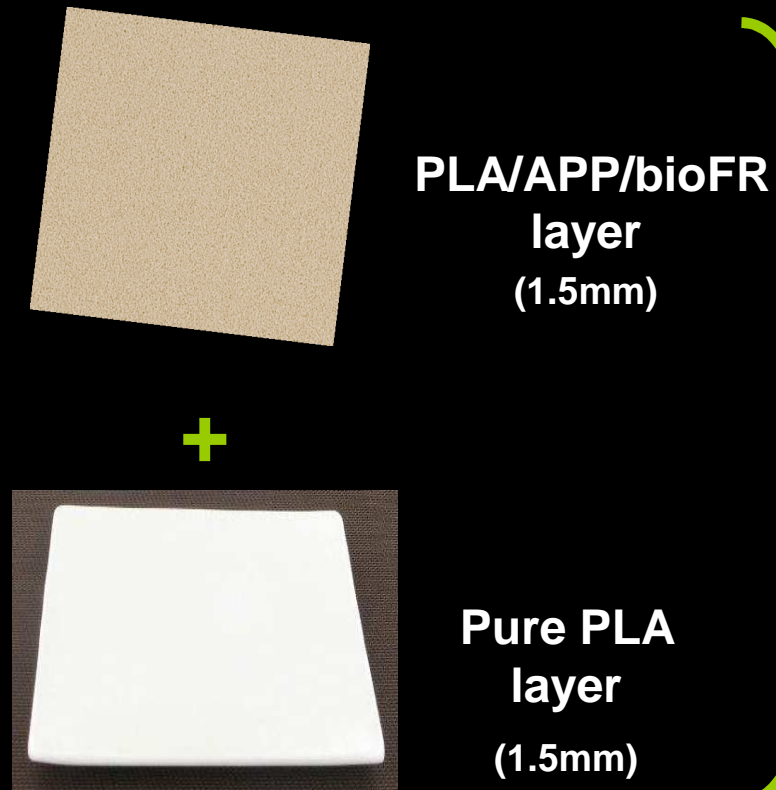
External heat flux = 35 kW/m²



Acceptable performance
via an intumescent
mechanism

Total loading = 40 wt.-%

Preparation of multilayers

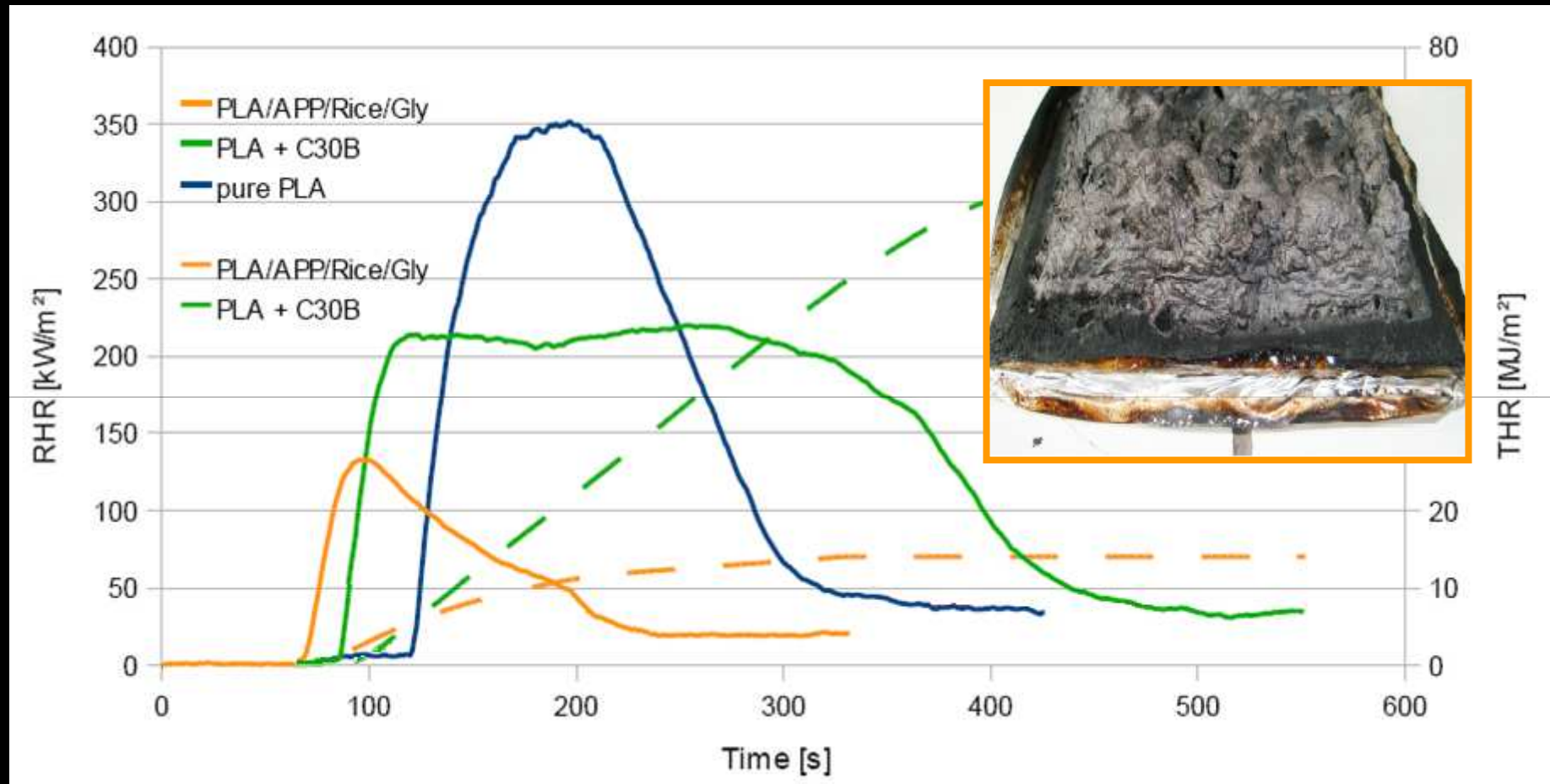


press

Multilayer

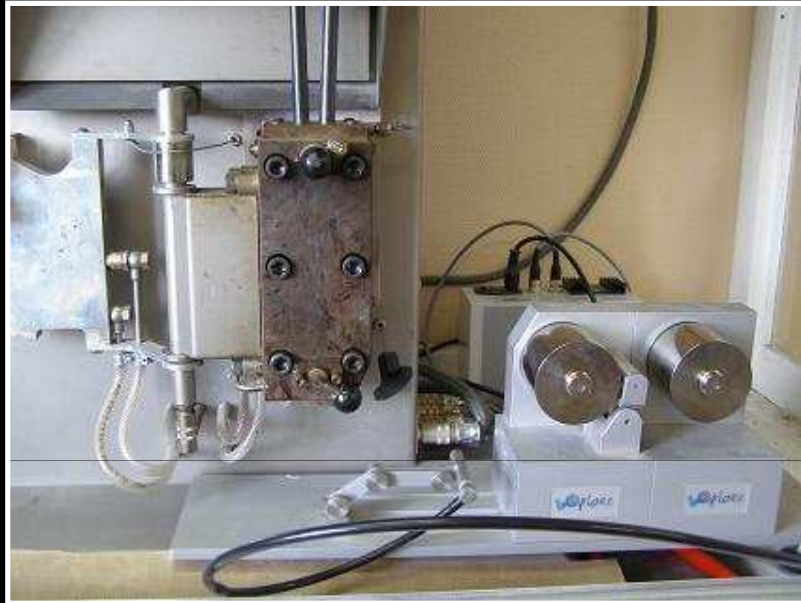
Aim : To decrease the quantity of additives

Fire Retardant Properties of multilayers

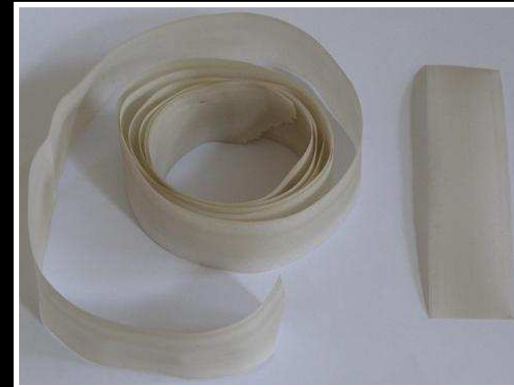


Large decrease of the pRHR and THR with starch

Preparation of materials



extruder



FILM

Thickness : 250µm



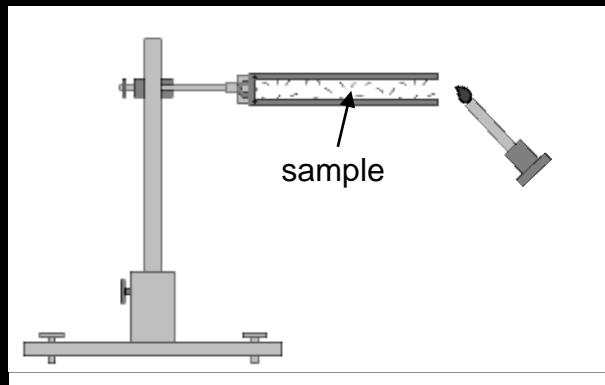
nonwoven



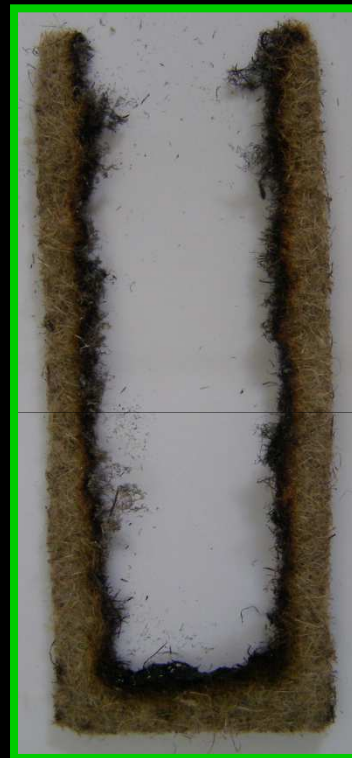
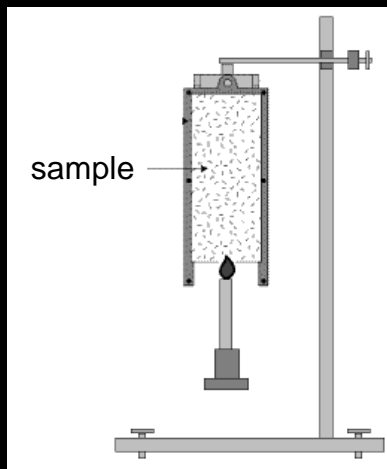
press



Fire Retardant Properties



Horizontal and vertical fire tests



Hemp
nonwoven



Nonwoven +
PLA film



Nonwoven +
intumescent film

Conclusions

- **Development of bio-based flame retardant PLA**
- **Use of bio-based char former in intumescent formulation: *high performance***
- **Use of 'full' bio-based intumescent formulation: *acceptable performance and promising approach***
- **Development of solutions for specific applications**

Co-workers and Acknowledgements

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