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# Formulation and characterizations of nanoenergetic compositions

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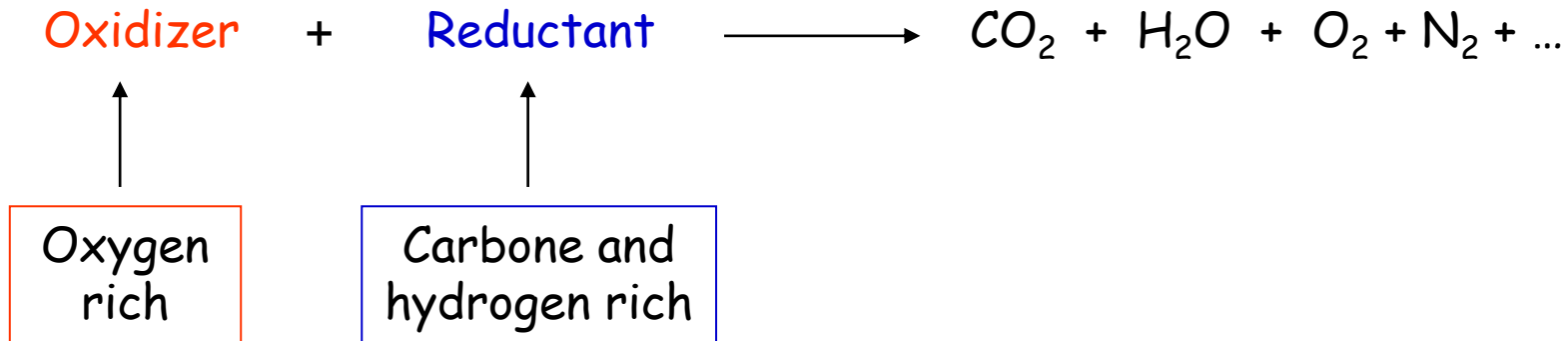
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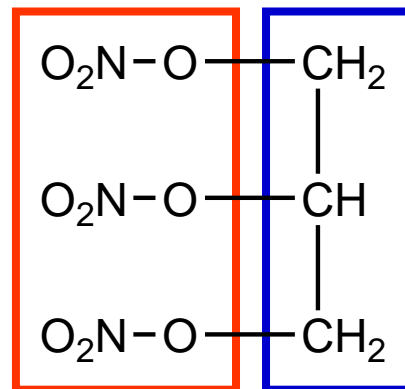
# Energetic materials and chemistry



Energetic material: mixture between an oxidizer and a reductant (molecule or composition). The decomposition reaction can be written as follow



Energetic molecule: unimolecular reaction



Oxidizer and reductant atoms are sufficiently close to one another for the decomposition reaction to take place 😊

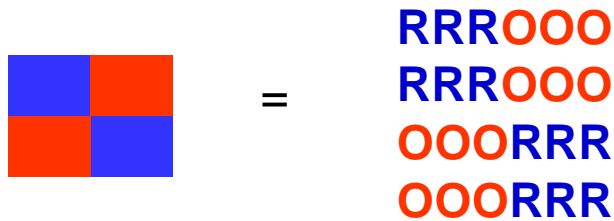
☹️ Drawback: synthetic feasibility

# Energetic materials and chemistry



Energetic macrocomposition: mixture of powders

Oxidizer powder (O) + reductant powder (R)



Oxidizer and reductant atoms are distant to one another and the decomposition reaction kinetic may be too slow 😞

Energetic nanocomposition: mixture of particles

## Advantages:

- homogeneity of the composition (sensitivity and performances)
- 😊 high contact area between O and R (performances)
- kinetic of decomposition reaction comparable to unimolecular decomposition reaction
- no problem of synthetic feasibility
- versatility of the composition (oxygen balance adjustment depending on the application)

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## Prepared and tested materials

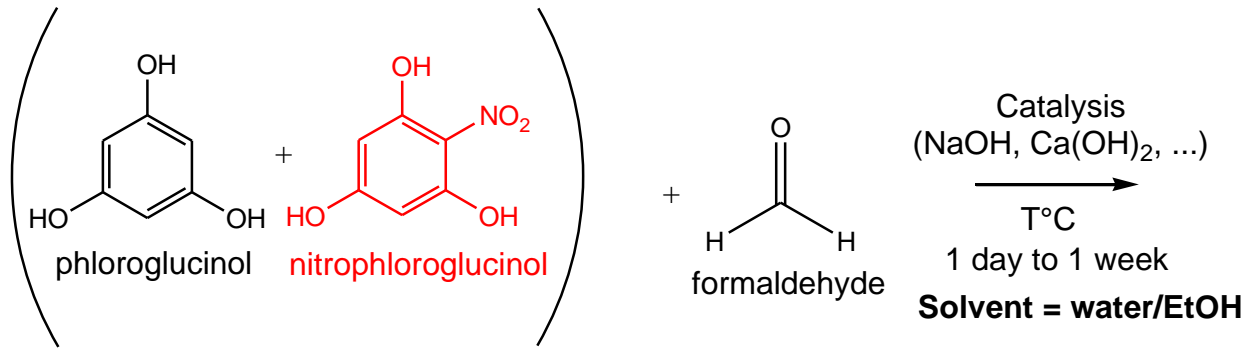
- ✓ Synthesis and formulation
- ✓ Physical characterizations
- ✓ Energetic results

## Conclusion and Prospects

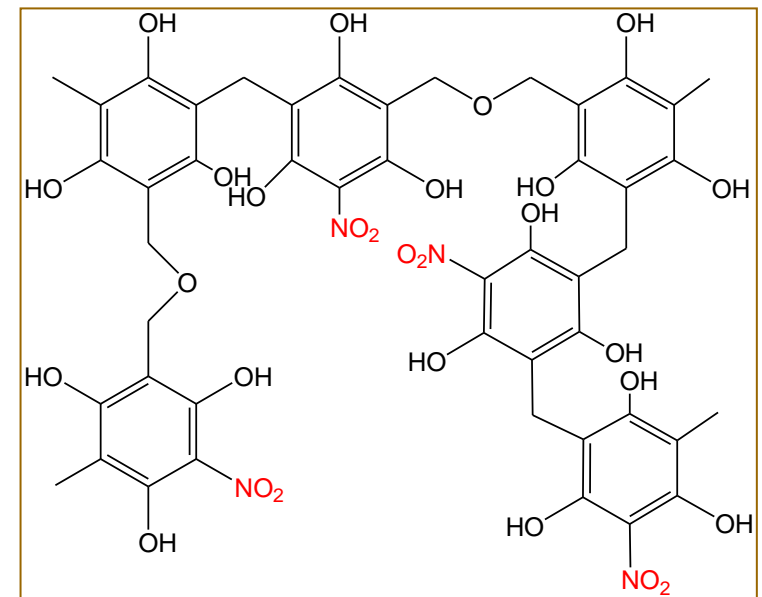
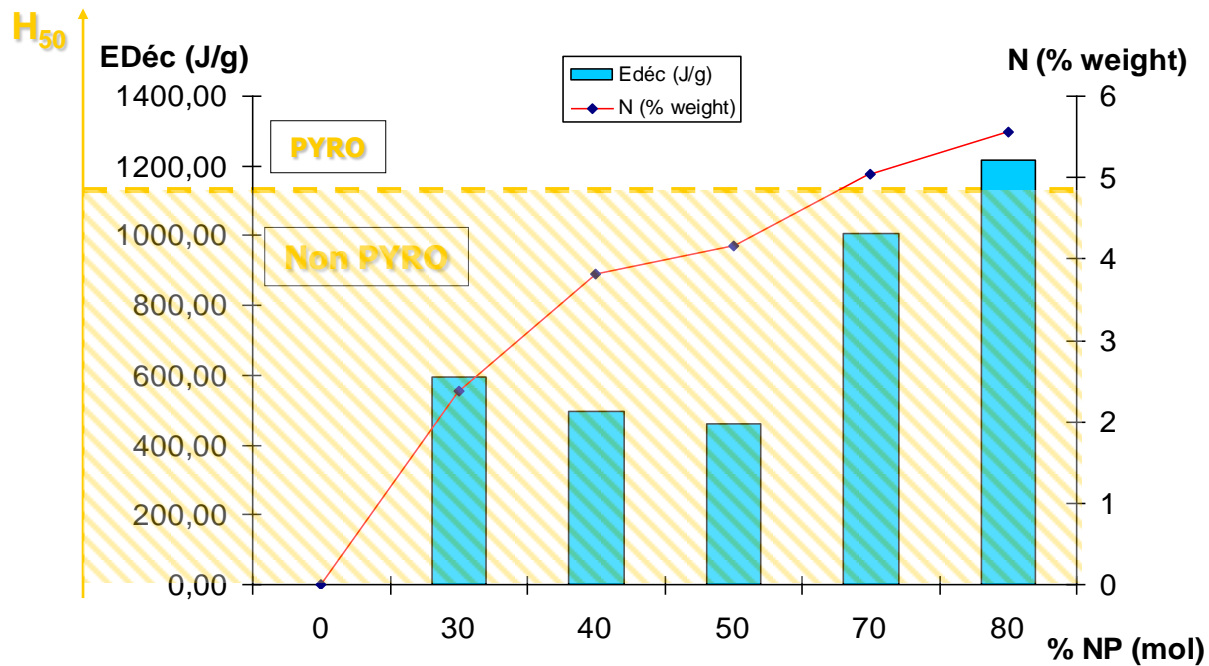
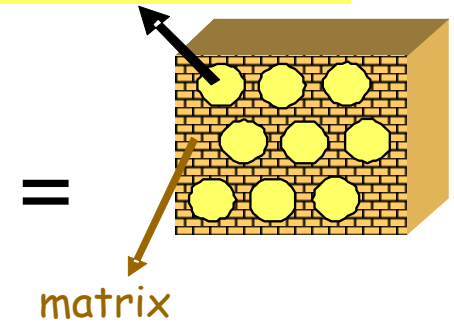
# Synthesis and formulation



## Synthesis of new functionalized organogels:



Solvent from the synthesis



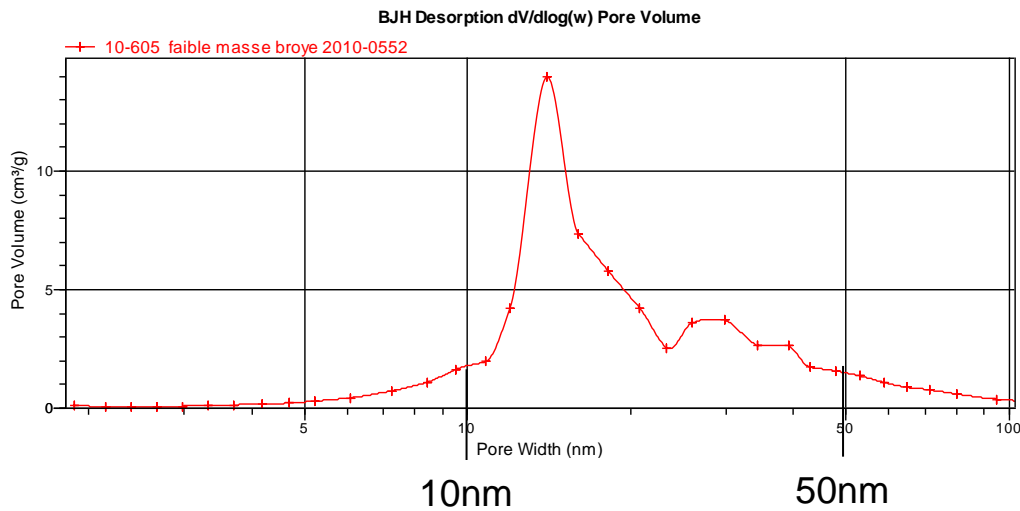
# Synthesis and formulation

✓ BET analysis of PF and (P/NP7/3)F aerogels (supercritical CO<sub>2</sub> drying)

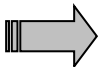
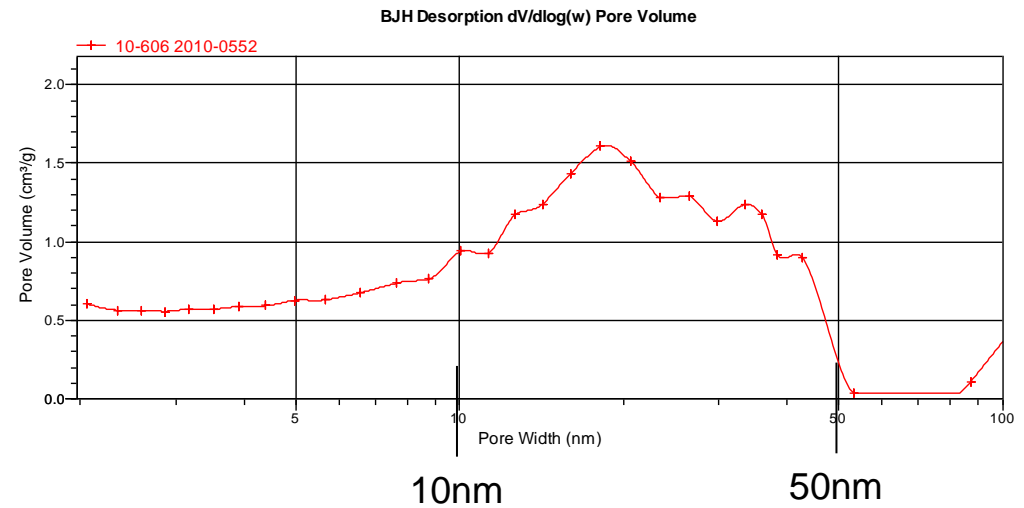


Sample	Specific area (m <sup>2</sup> /g)
PF	795 ± 40
(P/NP7/3)F	688 ± 40

PF gel : Pore size distribution



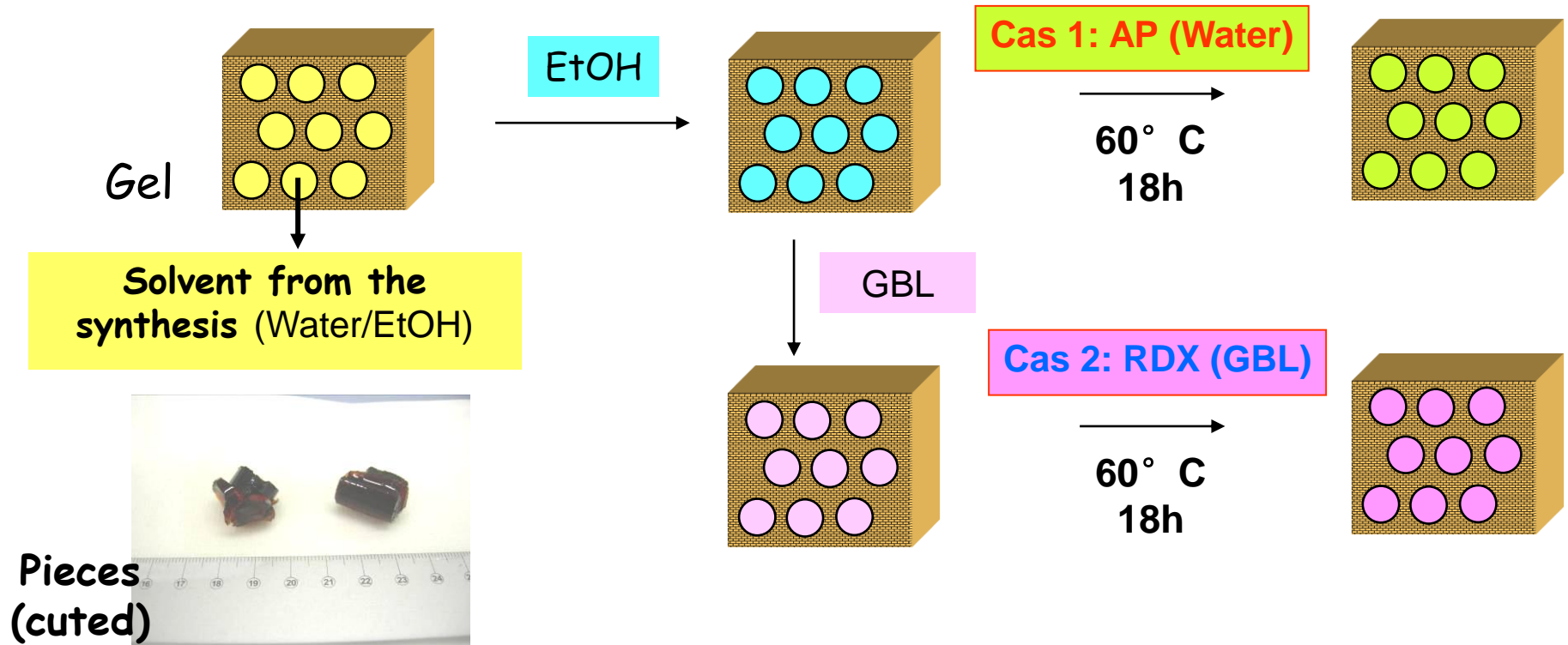
((P/NP7/3)F gel) : Pore size distribution



# Synthesis and formulation



Impregnation of an organogel with AP or RDX :

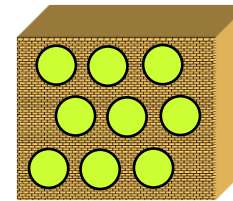


# Synthesis and formulation

From impregnated gel to powder :



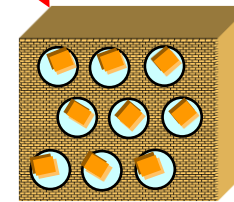
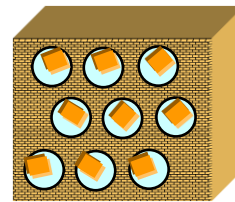
Impregnated gel



Crystallization of charge particles

Quenching EtOH  
(0 °C 2 h)

Cryotransfer  
(liquid nitrogen)



Drying and powdering

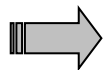
Wet mortar crushing  
+  
Normal drying  
(atmospheric P, ambient T)

Freeze drying  
+  
Dry crushing  
(vibratory ball mill)

Xerogel



Cryogel





# Physical characterizations of prepared materials

## X Ray powder Diffraction :

80 wt%. AP charge ( $BO_{CO_2} = 0\%$ )	Average size of AP particles
PF/AP xerogel	> 120 nm (calculus : 480 nm) (repeatability : OK)
PF/AP cryogel	150 nm

## Imaging :

- Good microscopic homogeneity for xerogels and cryogels (SEM)
- Exocrystallization for xerogels

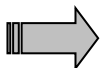
Before drying



After normal drying :

- White AP exocrystallites
- Partial destruction of the matrix

Example : xerogel balls

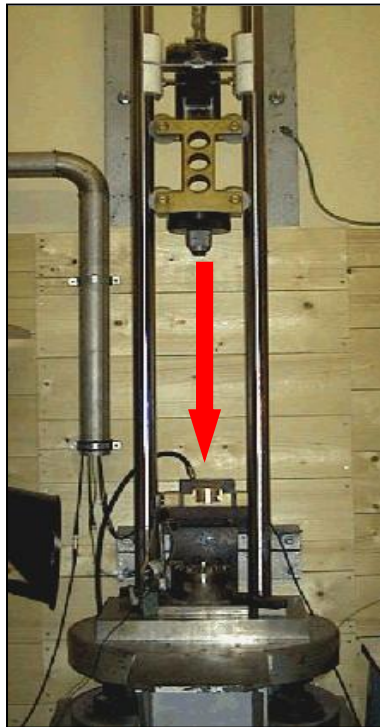


# Energetic characterizations : RDX as a charge



## ➤ Instrumented drop-weight apparatus :

RDX charge  
 $OB_{CO_2} \sim -50 \%$   
 $\sim 70$  to  $75$  wt%.



	Impact $H_{50}$ (mm)/ $P_{MAX}$ (bars)	
	Macro	Nano xerogel
RDX	100-150 / 5	/
PF/RDX	140 / 3,92	137 / 1,92 (- 51%)
P/NP(7/3)F/RDX	79 / 3,16	130 / 1,48 (- 53 %)
P/NP(3/7)F/RDX	95 / 3,63	/

Nanodispersions are slightly less sensitive and less powerful than mixture of powders

- ✓ Mixture of powders (macrosized) : matrix acts as a scraper
- ✓ Nanodispersions : matrix protects RDX towards aggression
- ✓ dispersion/dilution of RDX

Future work : increase the wt%. of RDX beyond 90 % → nanostructured and powerful intrinsic explosive

Difficulties with the xerogel way because probable destruction of the matrix during drying phase

➡ Cryogel process

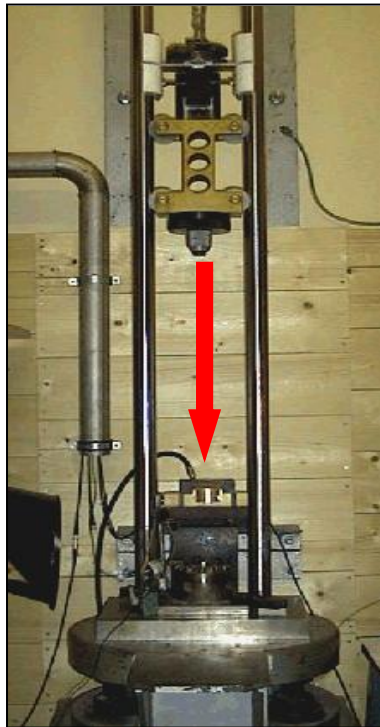
# Energetic characterizations : AP as a charge



## ➤ Instrumented drop-weight apparatus :

AP charge :  
 $OB_{CO_2} = 0\%$   
 $\sim 80wt\%$ .

	Impact $H_{50}$ (mm) / $P_{MAX}$ (bars)		
	Macro	Nano xerogel	Nano cryogel
PA	$\sim 500 / 0,75$	/	/
PF/PA	350 / 1,48	367 / 1,92 (+30%)	503 / 3,03 (+104%)
P/NP(7/3)F/PA	231 / 1,06	580 / 2,47 (+133%)	500-700/2 to 3,3 (+100 to 200%)
P/NP(3/7)F/PA	189 / 1,60	203 / 1,64 (+2%)	257 / 2,41 (+50%)



nanodispersions are less sensitive and more powerful than mixture of powders

- ✓ Same mechanical influence of the matrix than for RDX compositions
- ✓ Better mix between oxidizer and reductant when nanodispersed
- ✓ Cryogel way better than xerogel way

➡ Cryogel process

# Energetic characterizations on AP cryogels

➤ Closed-chamber combustion :

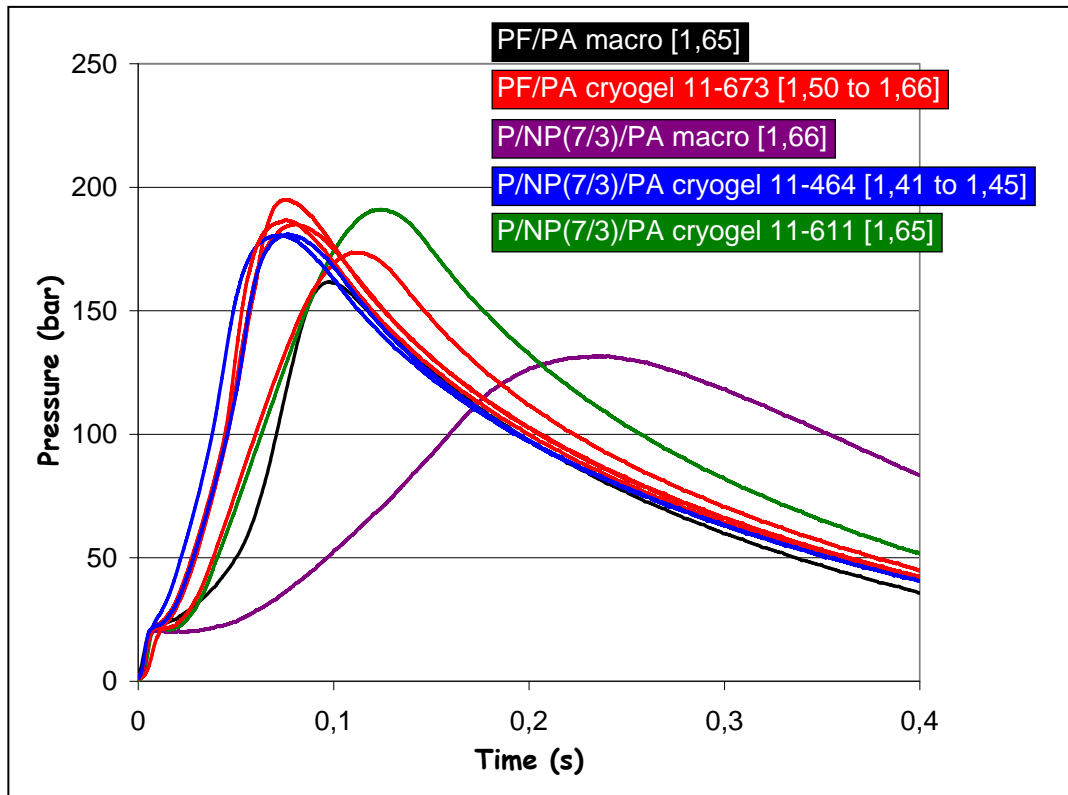
2 g pellets in a 64 cm<sup>3</sup> chamber



Pressing sequence : 60° C + 3x1000 bar)



$\rho = 1,65 \text{ g/cm}^3$



Scatter of pellets densities due to:

- ✓ Microscopic differences from batch to batch (freeze-drying, crushing conditions, ...)
- ✓ Conservation conditions,
- ✓ ...



Systematic deconsolidative burning for low density pellets

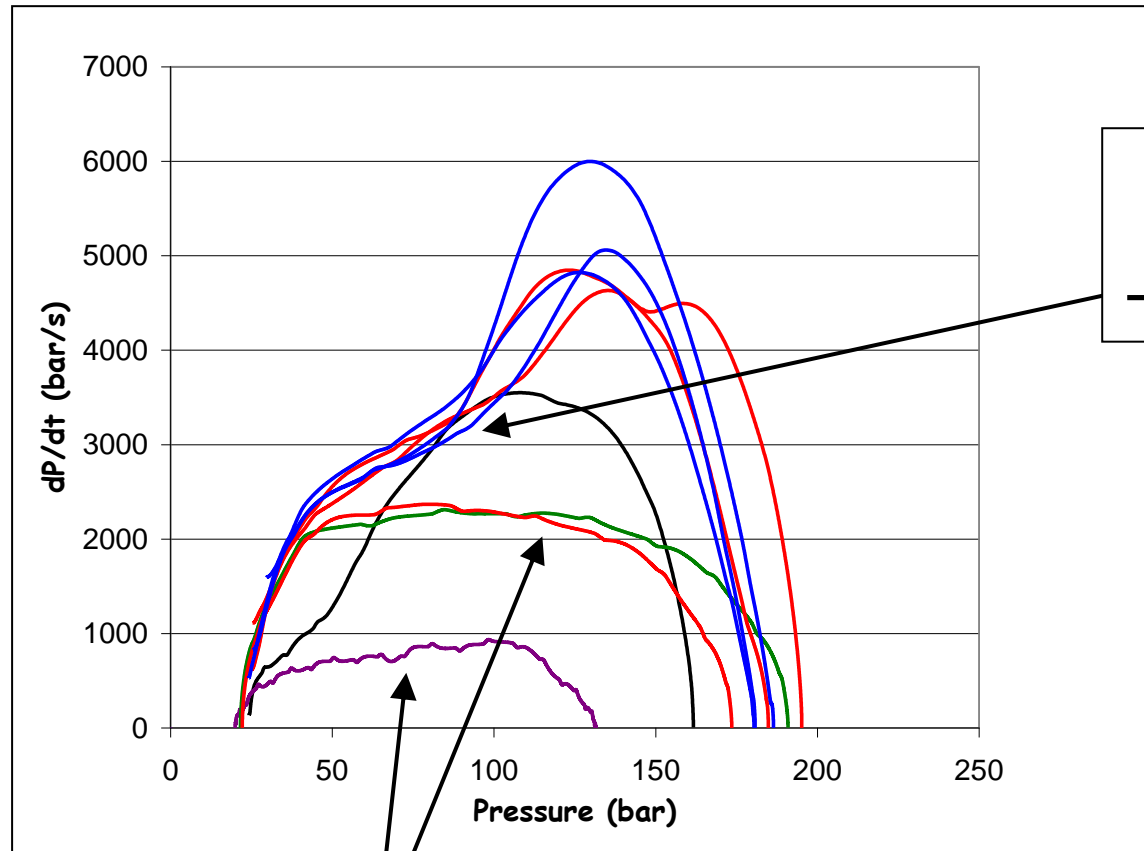


Difficulties to compare and to discriminate the different formulations

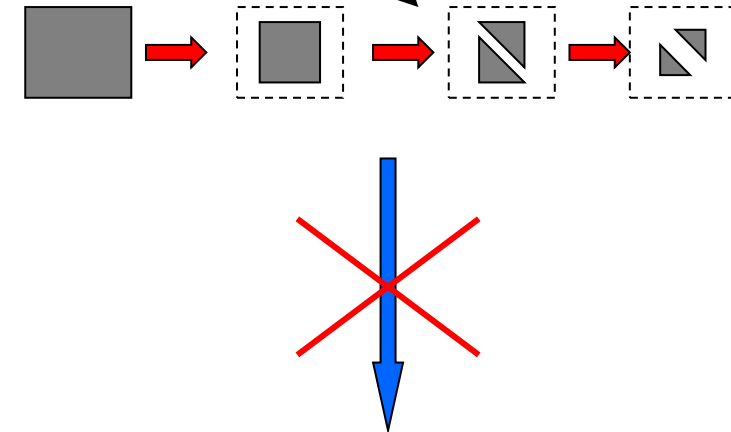
# Energetic characterizations on AP cryogels



➤ Closed-chamber combustion :



Slope break-up of the pressurisation speed  
→ Deconsolidative burning



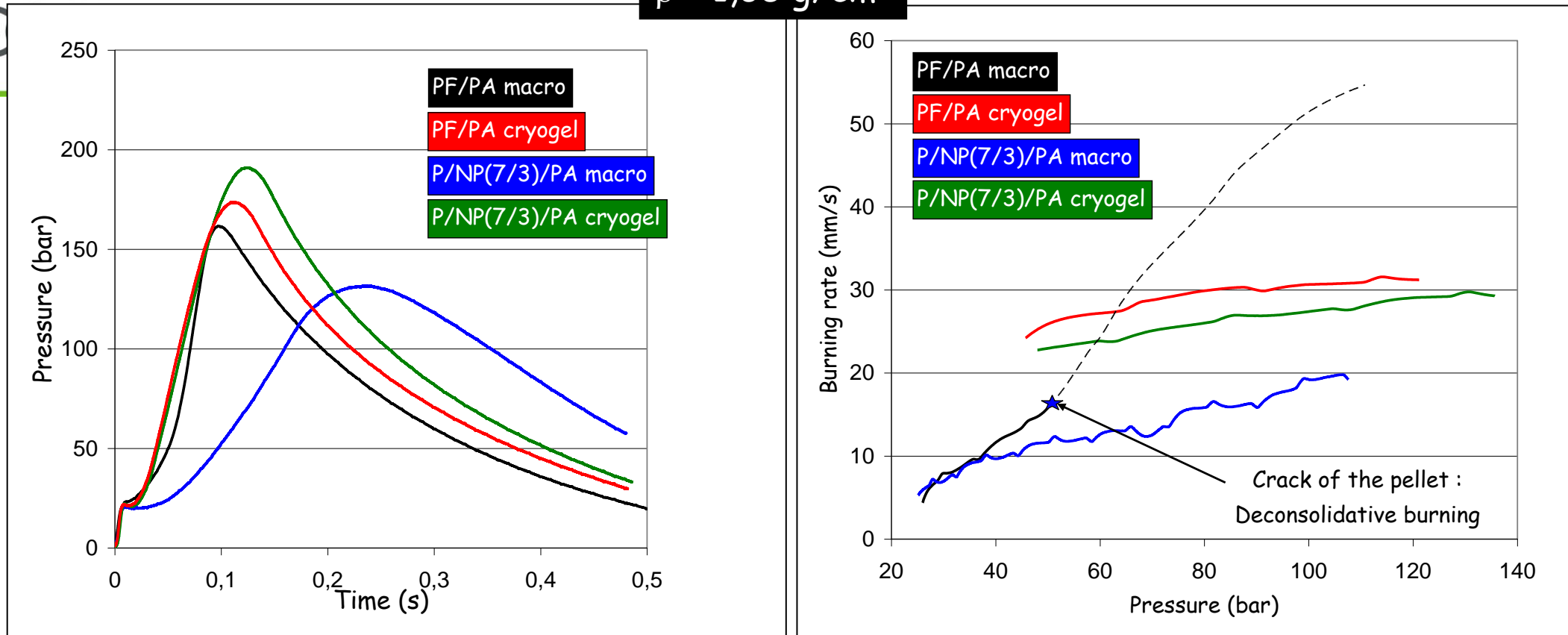
Bell-shaped curves → Regular burning



Combustion rate

# Energetic characterizations on AP cryogels

$\rho = 1,65 \text{ g/cm}^3$



- Burning rate of the nanosized formulations is about two or three times higher than the one of the mixtures of powders
- Nanostructuration guarantees a stable combustion all over the explored pressure range (exponent pressure < 1)

# Conclusion

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- New energetic functionalized organogels have been synthesized and their ability to constrain the charge to nanostructure has been illustrated.
- Nanodispersions tend to be less sensitive than mixture of powders (Impact sensitivity).
- When they decompose, AP based nanomaterials are more powerful than mixture of powders.
- Combustion of nanodispersions shows improved propulsion performances (burning rate and combustion stability) compared to mixture of powders.
- A scale-up of the cryogel process has recently been done with success, allowing us to produce batches of 125 g of nanoformulations  
→ Use in propellant formulation

# Prospects

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- Reproducibility of the cryogel process to nanostructure is demonstrated but improvements must be done to control and/or to tune the microstructure
- Ability of the cryogel process to nanostructure an intrinsic explosive (RDX) with charge ratio  $> 90$  wt%. → nanostructured powerful intrinsic explosive.

THANK YOU FOR YOUR ATTENTION





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